US007966866B2

(12) **United States Patent**
Hansma et al.

(10) **Patent No.:** **US 7,966,866 B2**
(45) **Date of Patent:** **Jun. 28, 2011**

(54) **METHODS AND INSTRUMENTS FOR MATERIALS TESTING**

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 591 days.

(21) Appl. No.: **12/079,444**

(22) Filed: **Mar. 27, 2008**

(65) **Prior Publication Data**

US 2009/0056427 A1 Mar. 5, 2009

Related U.S. Application Data

(60) Provisional application No. 60/921,788, filed on Apr. 3, 2007.

(51) **Int. Cl.**
G01N 3/38 (2006.01)
G01N 3/317 (2006.01)
(52) **U.S. Cl.** **73/81**
(58) **Field of Classification Search** 73/81–85; 600/562
See application file for complete search history.

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(74) *Attorney, Agent, or Firm* — Vista IP Law Group LLP

(57) **ABSTRACT**

Methods and instruments for characterizing a material, such as the properties of bone in a living human subject, using a test probe constructed for insertion into the material and a reference probe aligned with the test probe in a housing. The housing is hand held or placed so that the reference probe contacts the surface of the material under pressure applied either by hand or by the weight of the housing. The test probe is inserted into the material to indent the material while maintaining the reference probe substantially under the hand pressure or weight of the housing allowing evaluation of a property of the material related to indentation of the material by the probe. Force can be generated by a voice coil in a magnet structure to the end of which the test probe is connected and supported in the magnet structure by a flexure, opposing flexures, a linear translation stage, or a linear bearing. Optionally, a measurement unit containing the test probe and reference probe is connected to a base unit with a wireless connection, allowing in the field material testing.

7 Claims, 27 Drawing Sheets
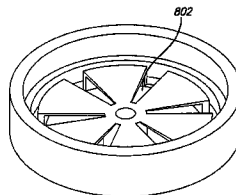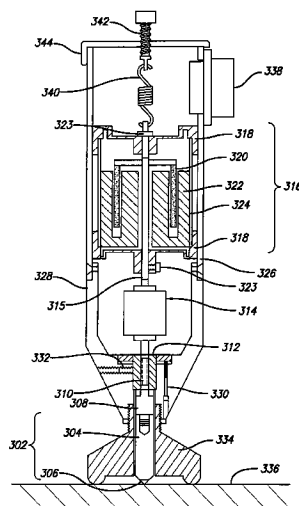
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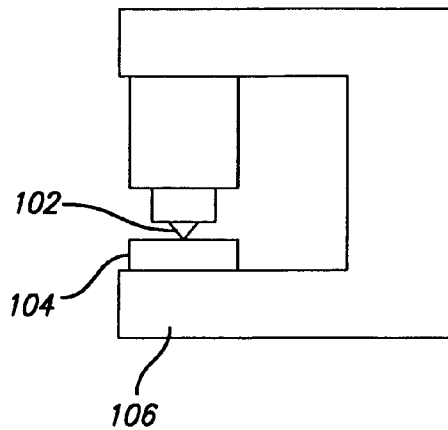
* cited by examiner
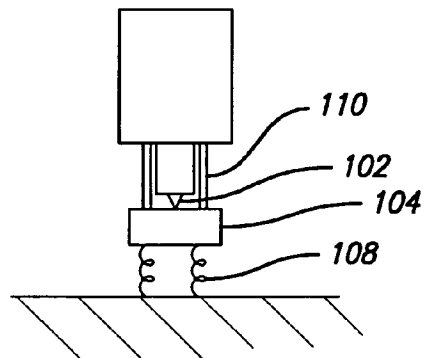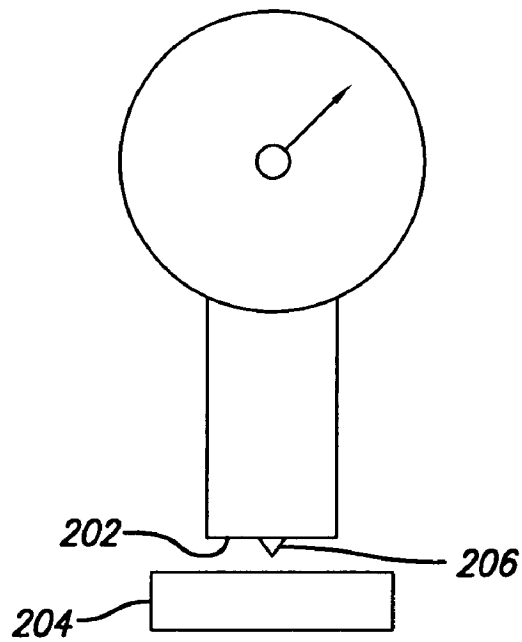
102
104
106

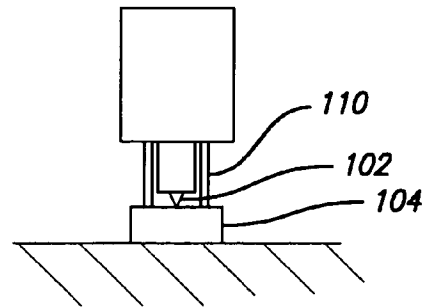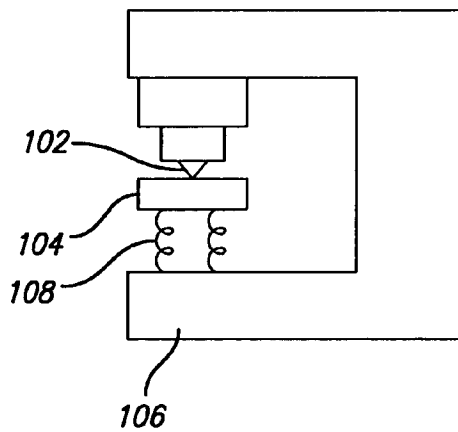*FIG. 1A*
PRIOR ART

110
102
104

FIG. 1B

102
104
108
106

FIG. 1C
PRIOR ART

110
102
104
108

FIG. 1D

FIG. 2A
PRIOR ART

202
206
204

208
210
202
206
204

FIG. 2B

FIG. 3

FIG. 4

511

510

514

512

508

509

501

500

506

504

502

516

518

503

FIG. 5A

FIG. 5B

602

FIG. 6A

6C   602   6C

604

606

FIG. 6B

606   602

604

FIG. 6C

TOTAL DEFORMATION
X 1e−2m

0.111
0.099
0.086
0.074
0.062
0.049
0.037
0.025
0.012
0.000

MIN

MAX

0   0.0088   0.018   0.027 (m)

FIG. 7

802

FIG. 8A

802

8C   8C

FIG. 8B   FIG. 8C

900

900

FIG. 9A

902

902

FIG. 9B

904

904   906

FIG. 9C

908   910

908   912   910

FIG. 9D

914

FIG. 9E

FIG. 10
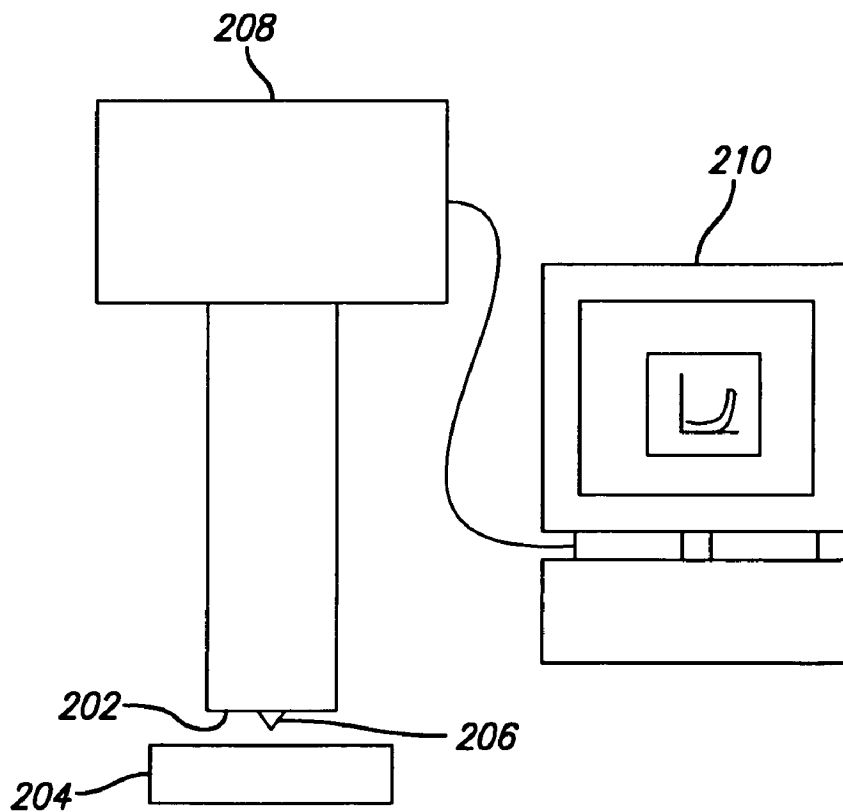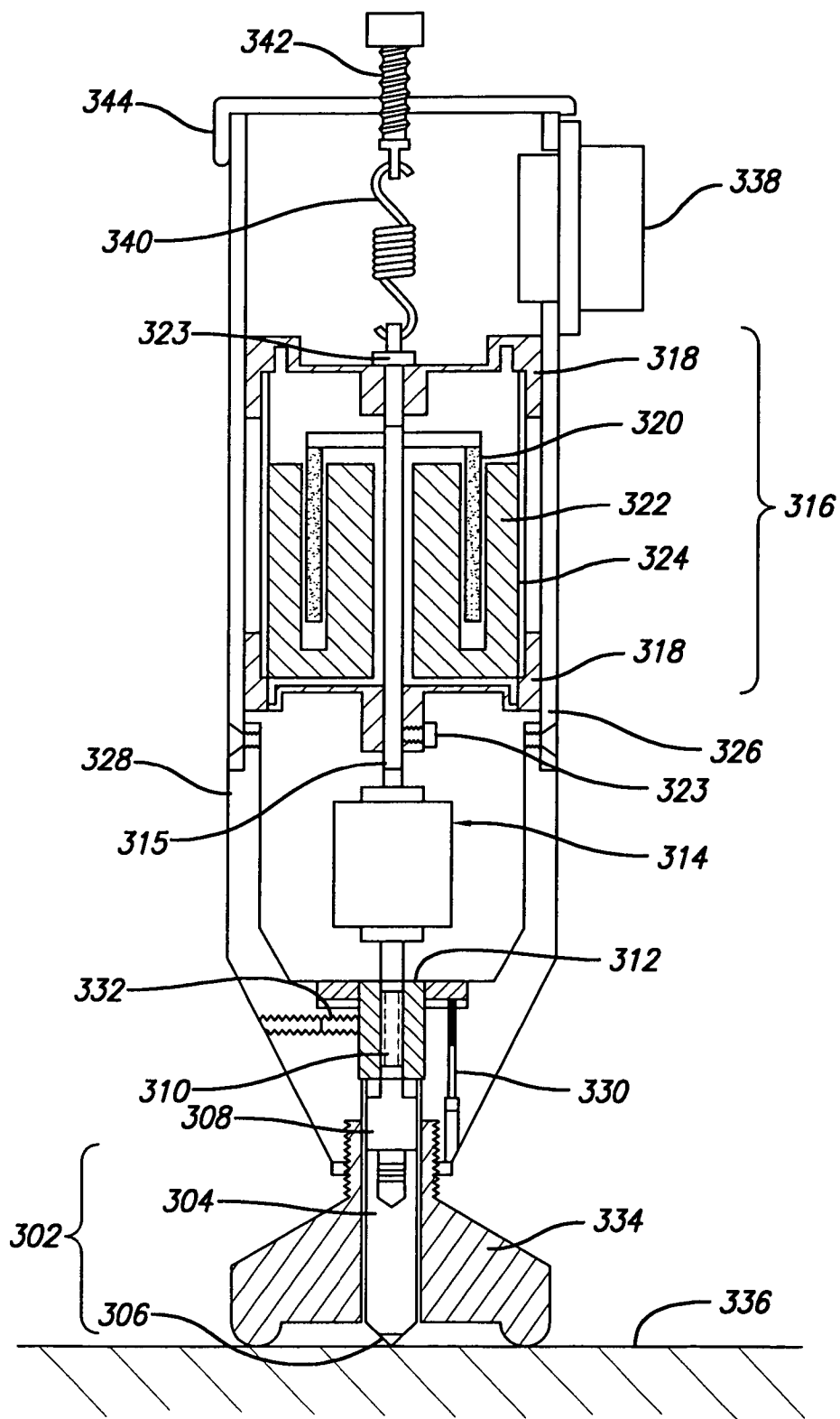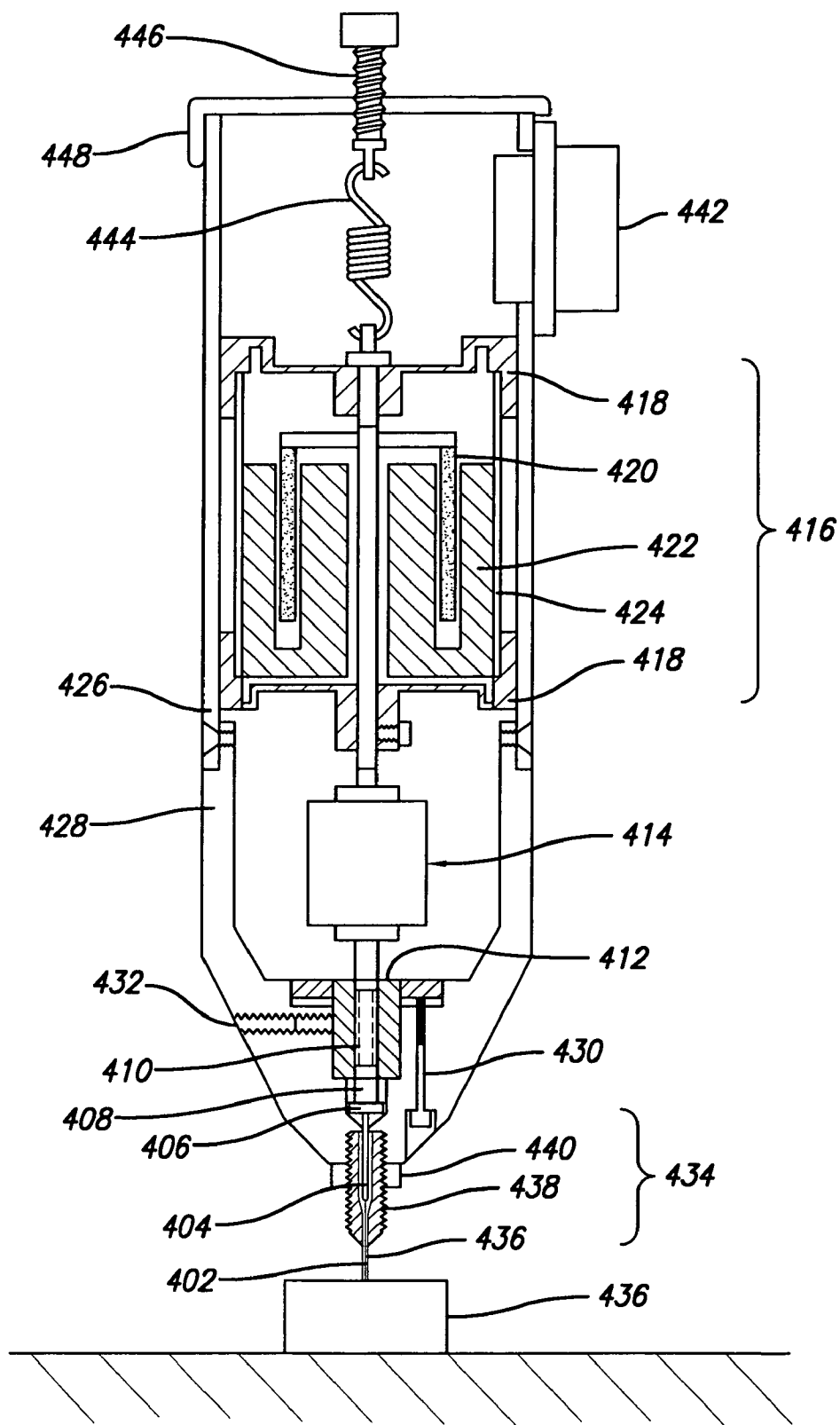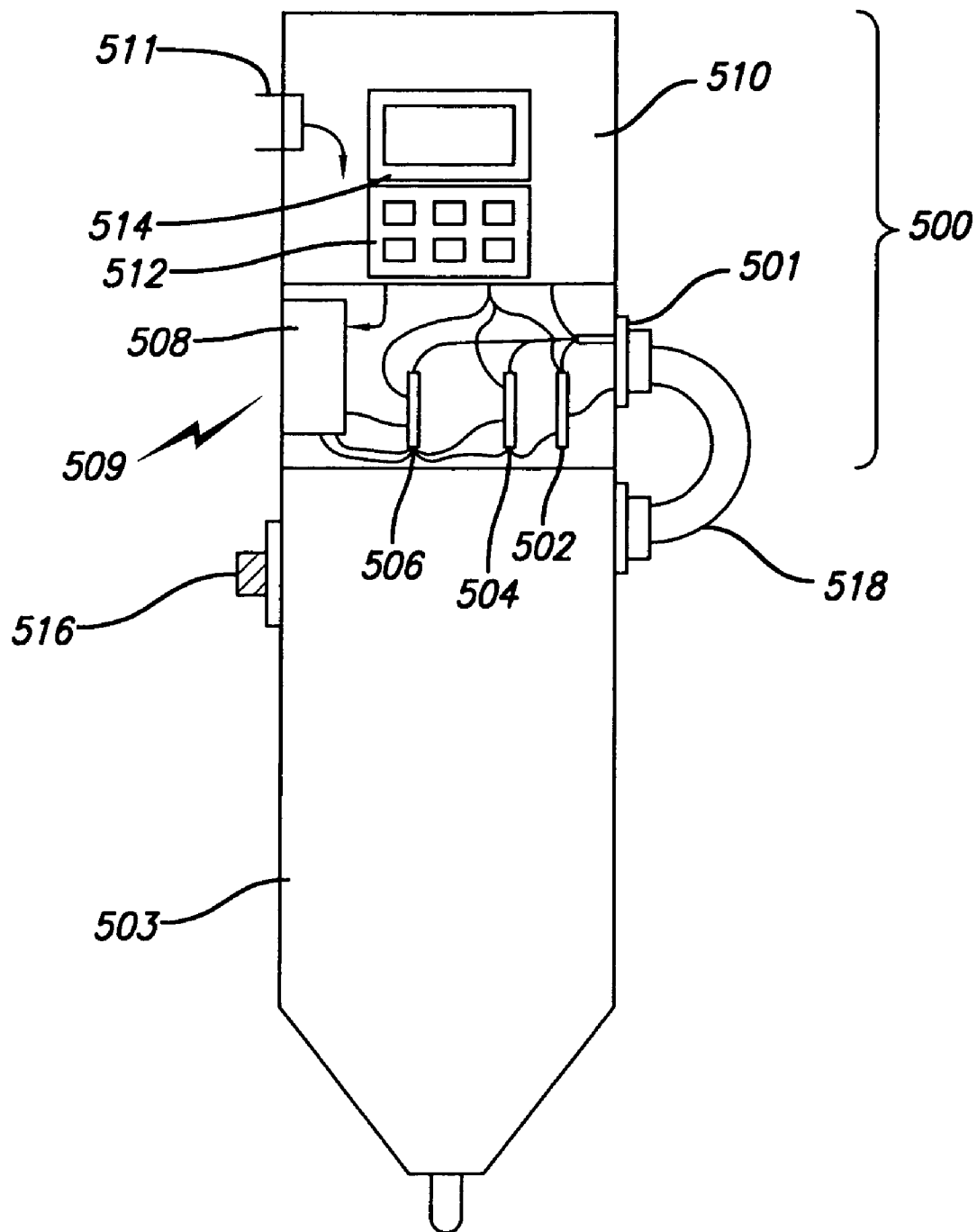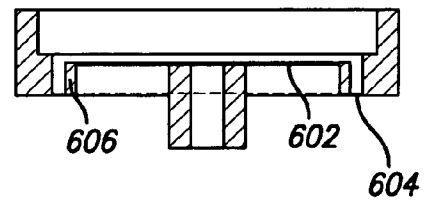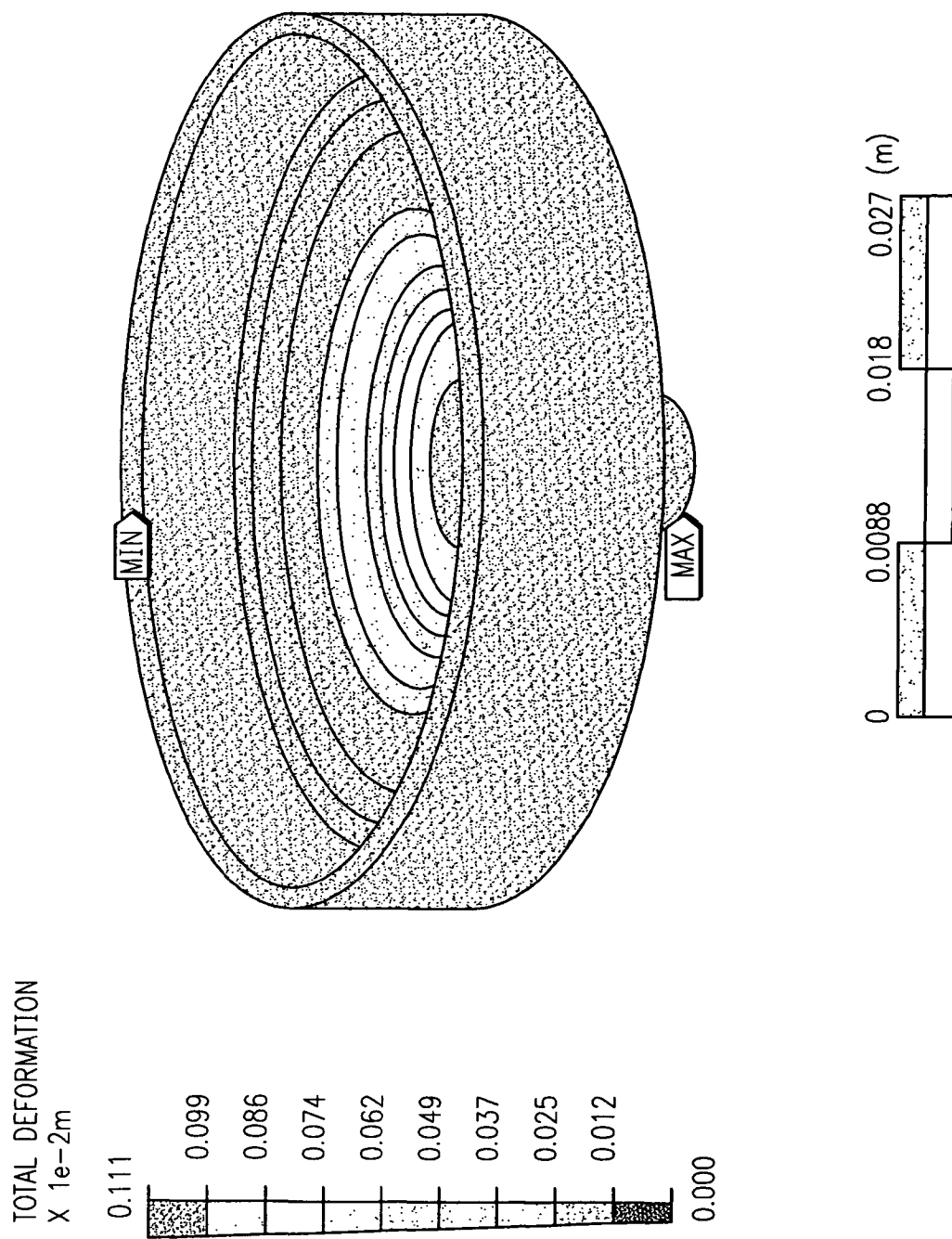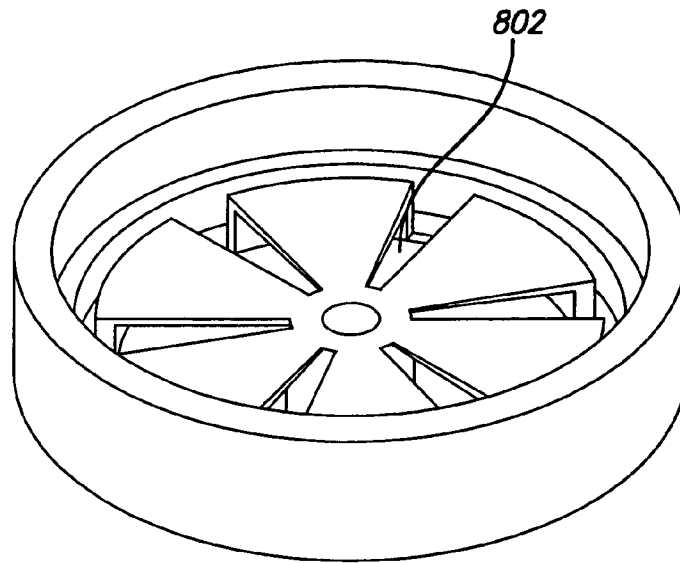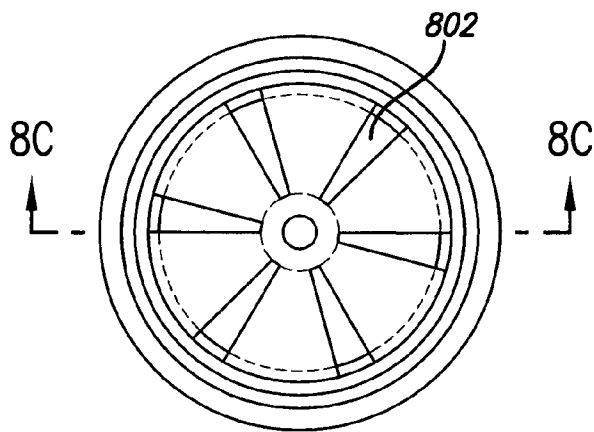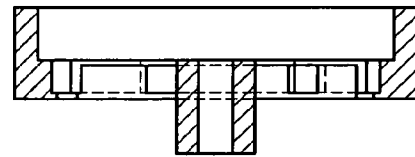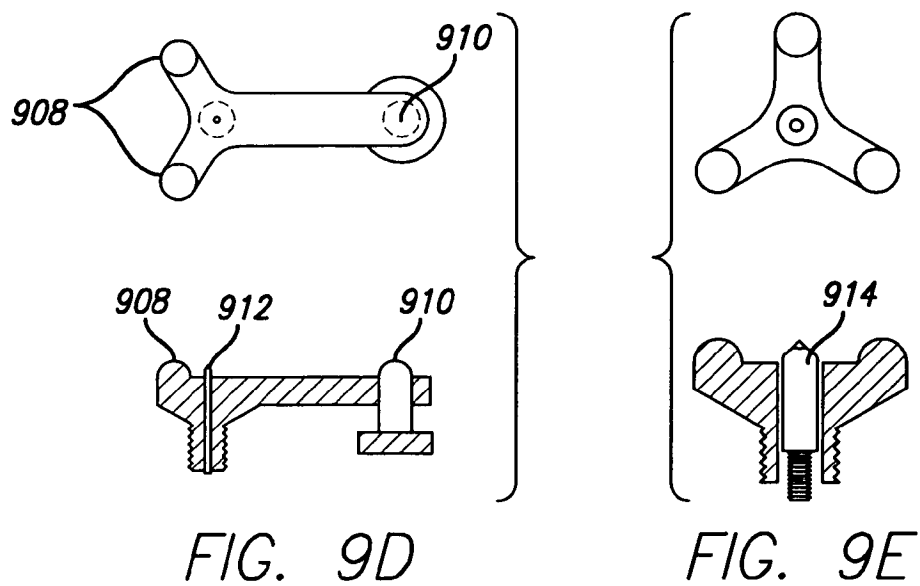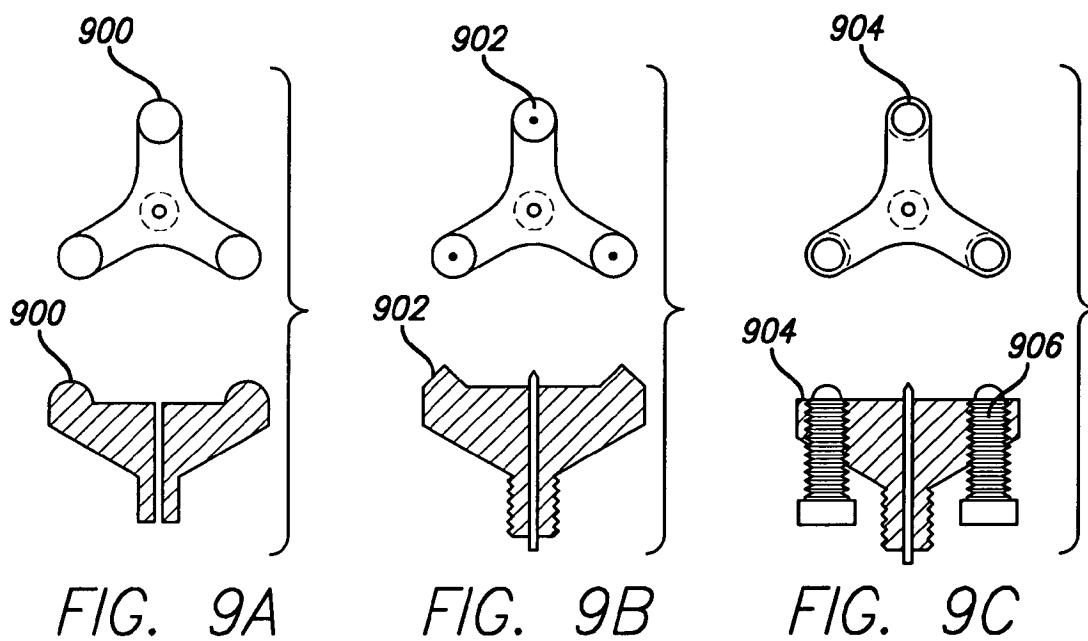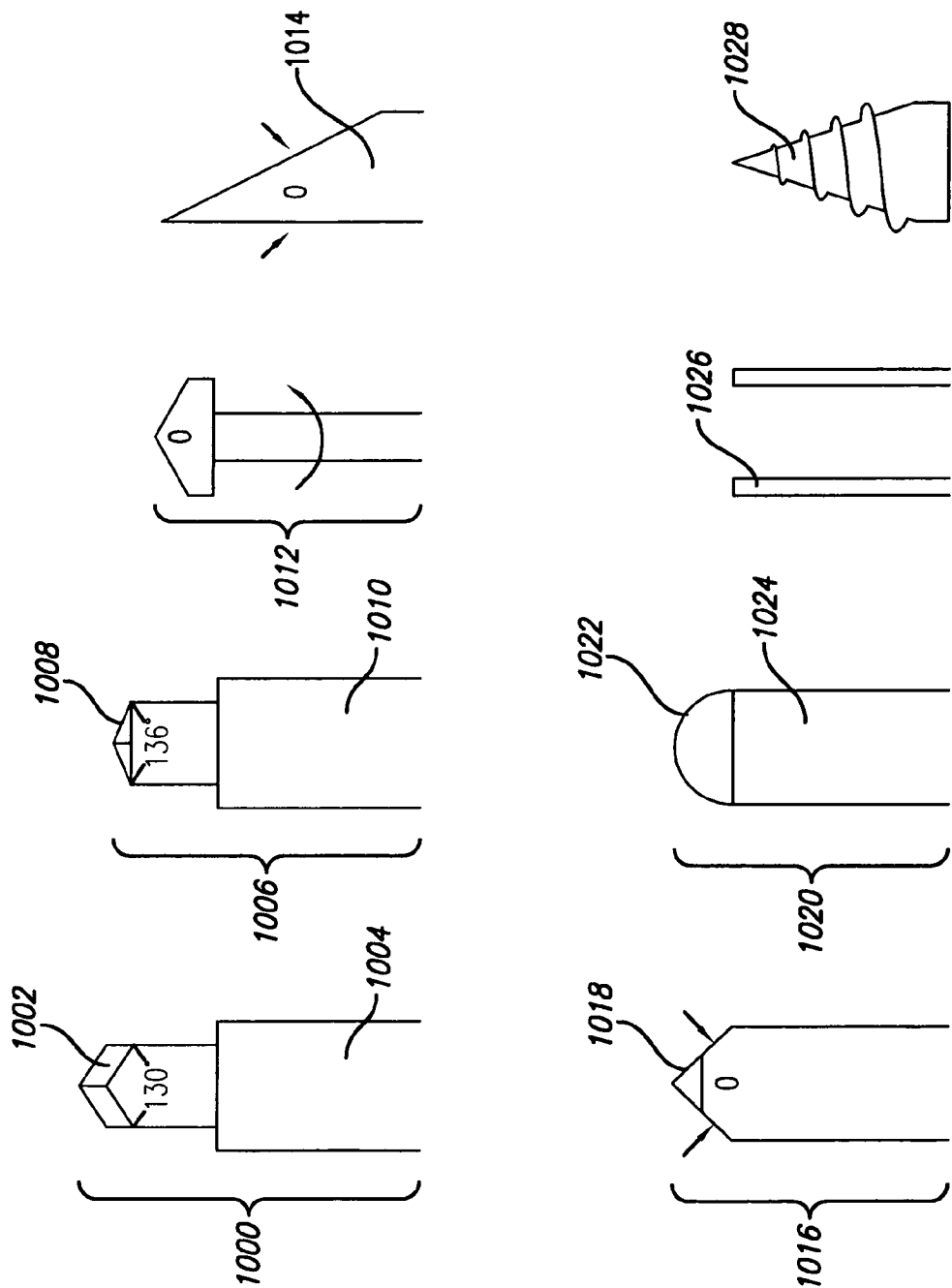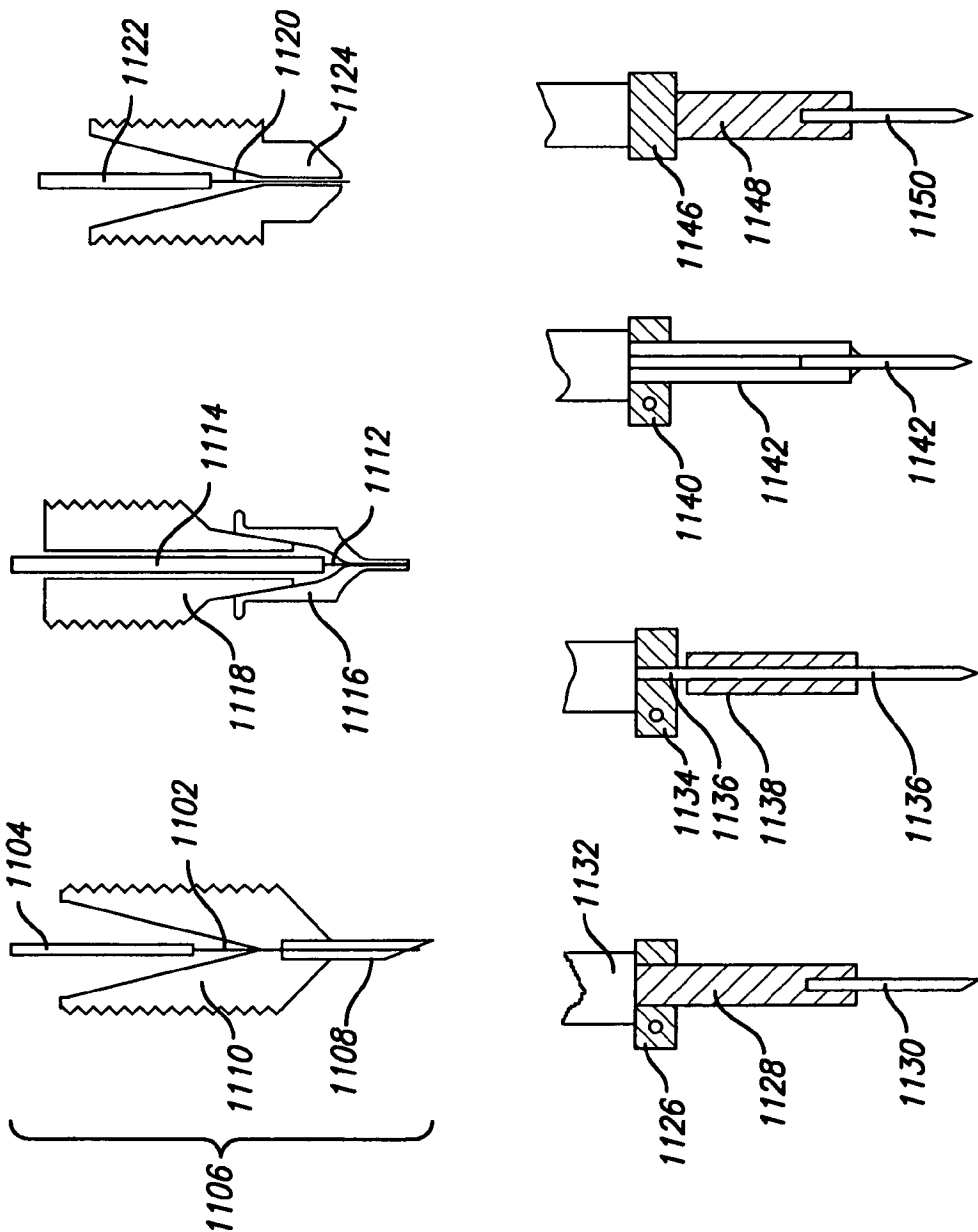
FIG. 11
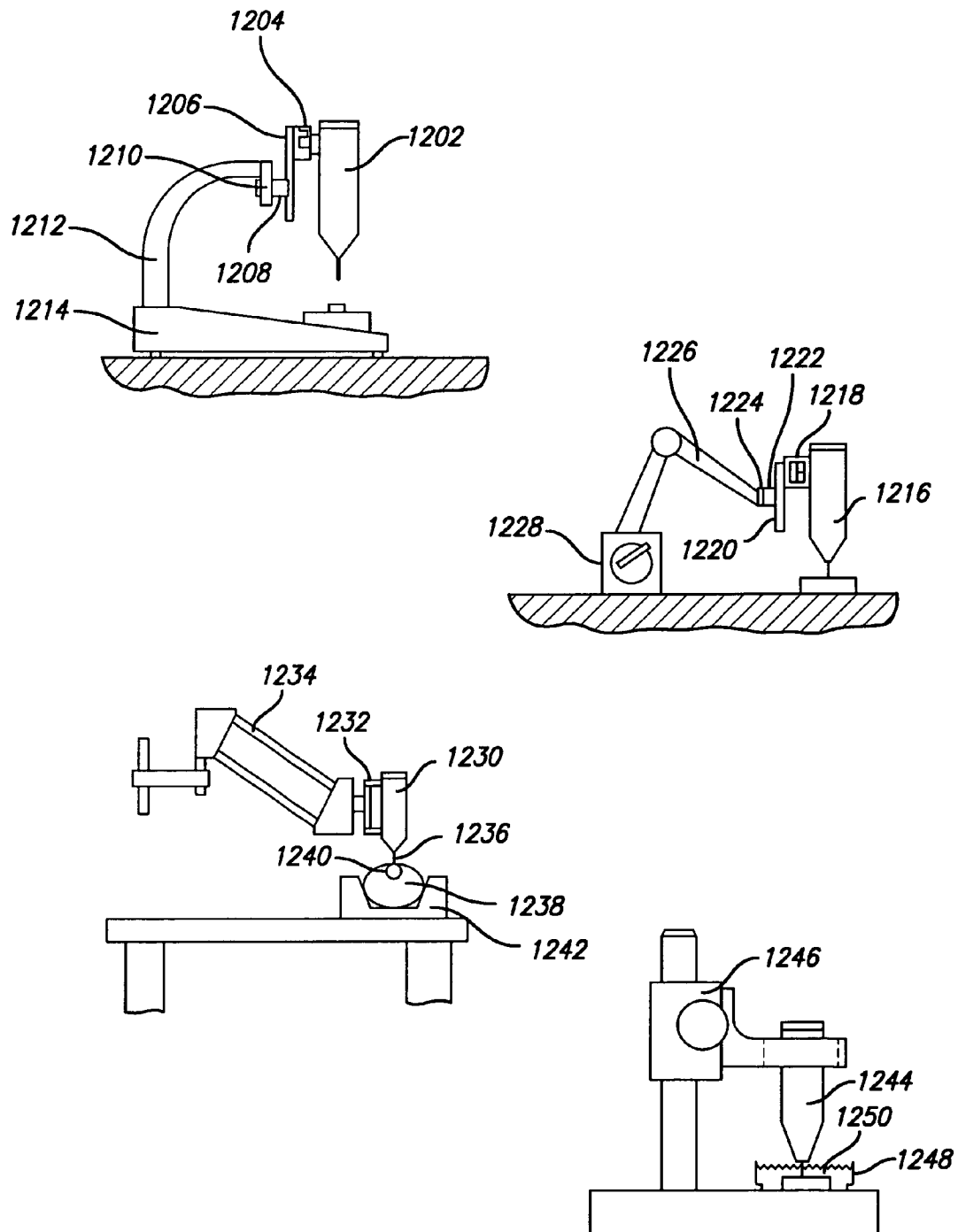
FIG. 12

FIG. 13

FIG. 14A

FIG. 14B

1506
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1512
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1508
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1518
1502
1504
1536
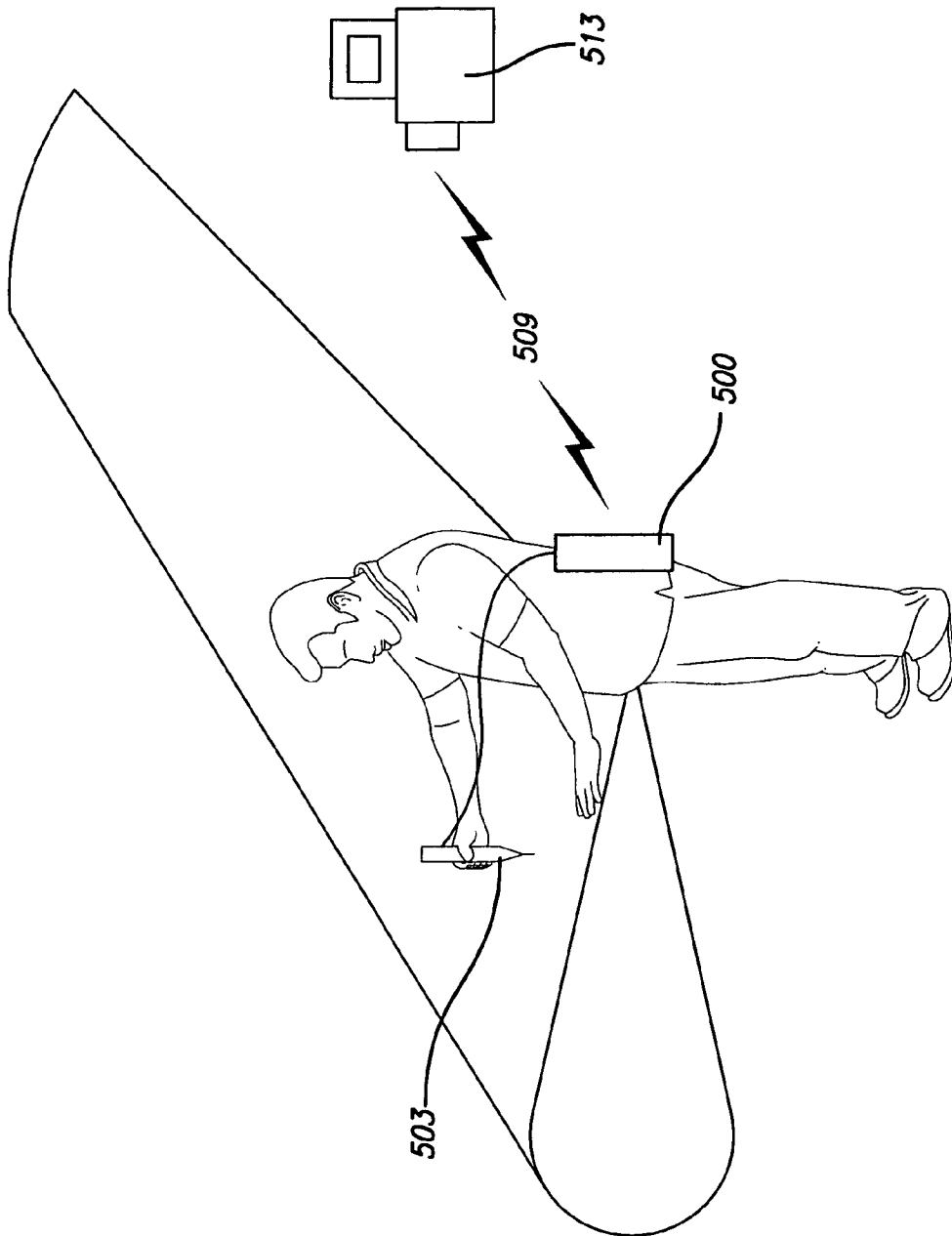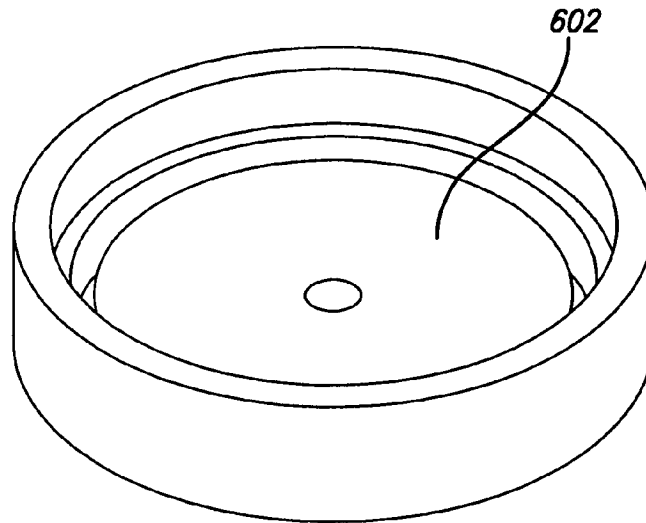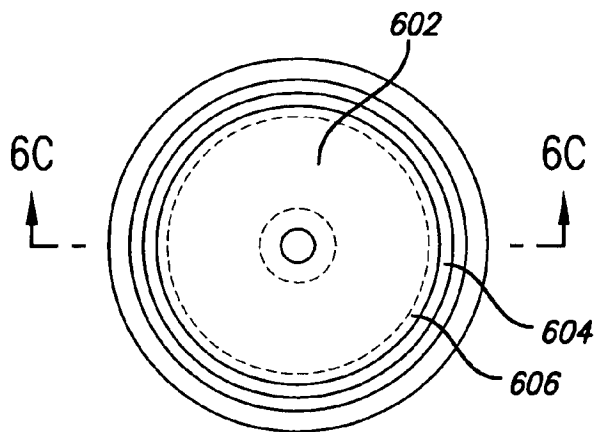1534
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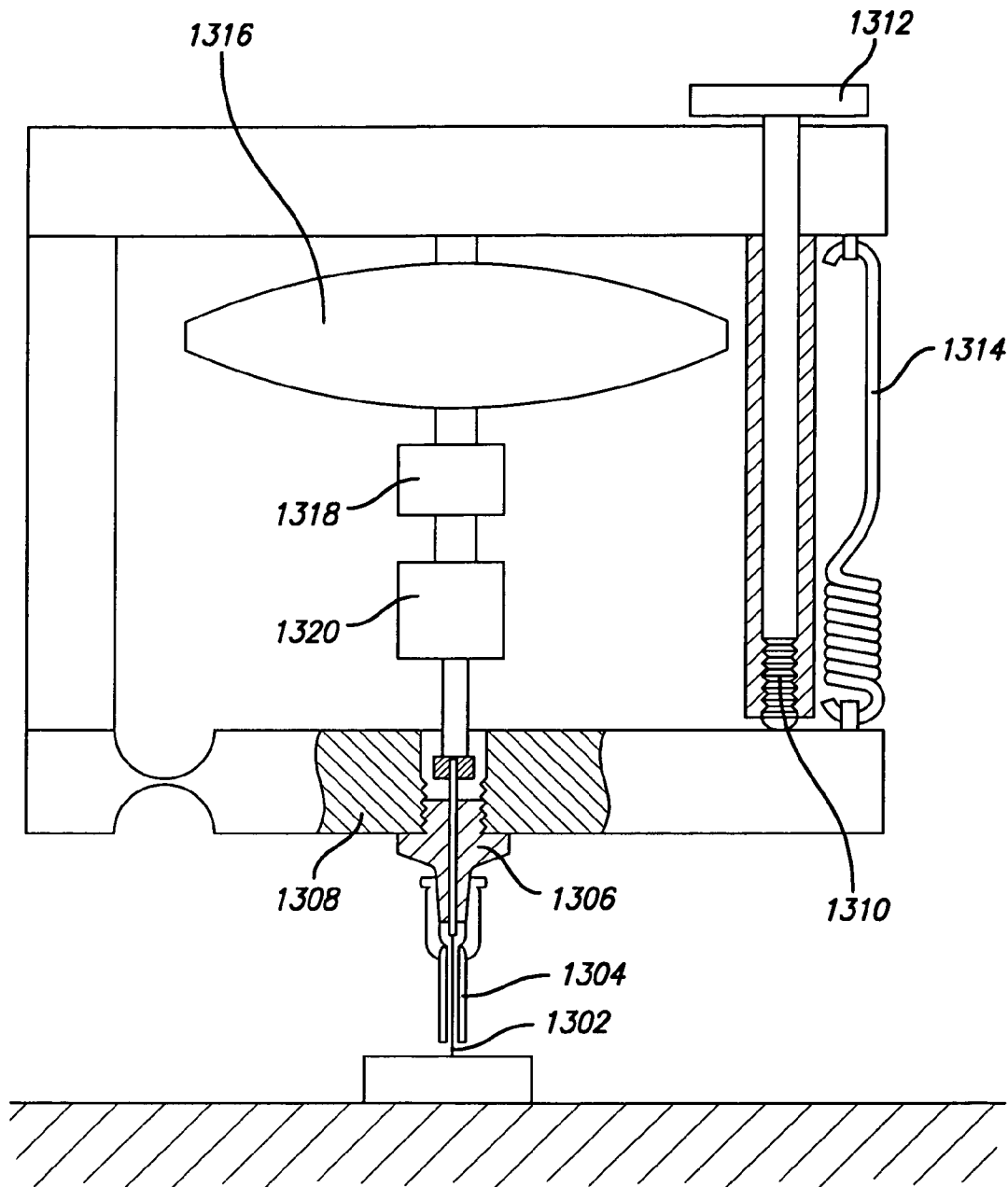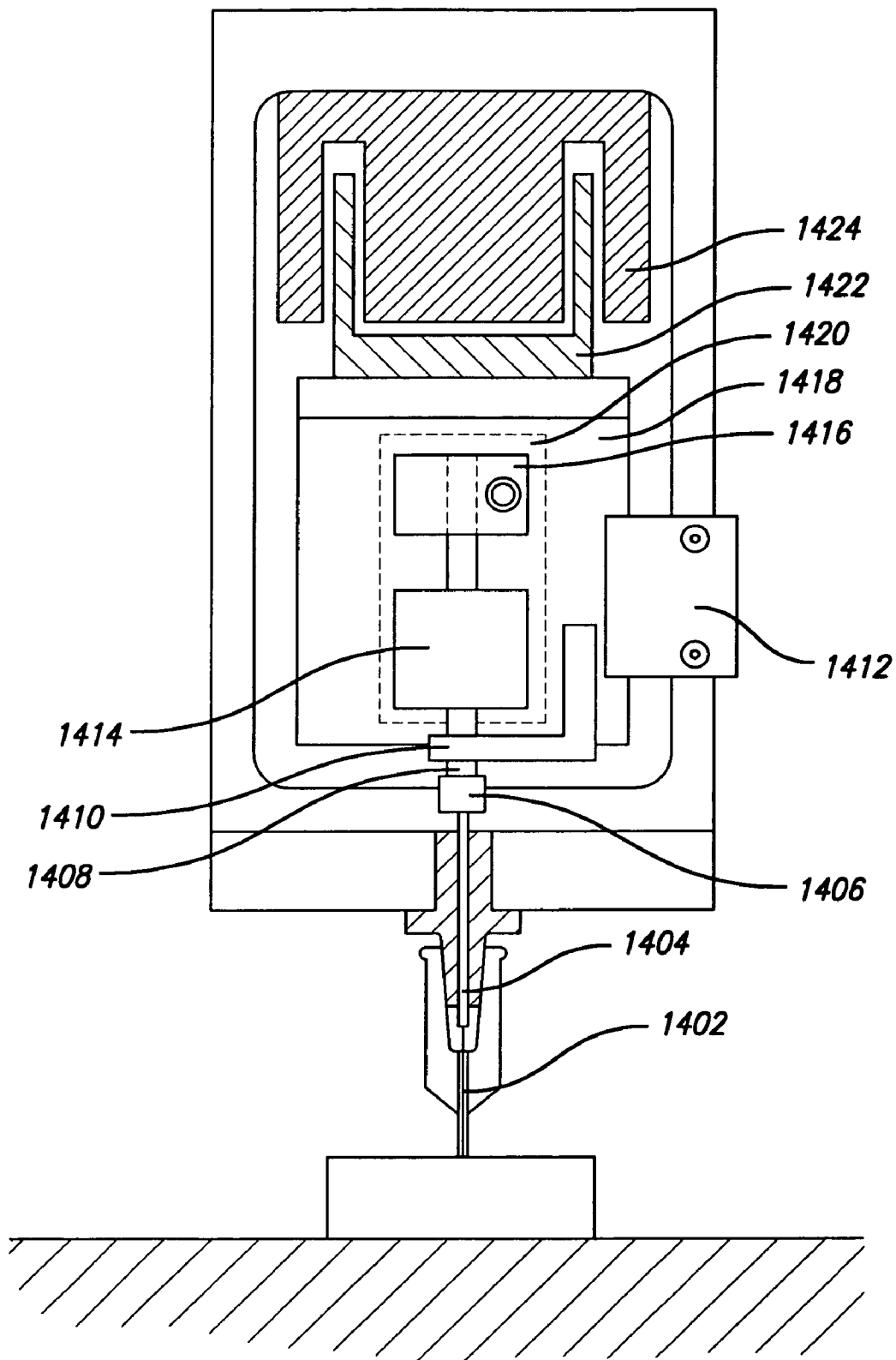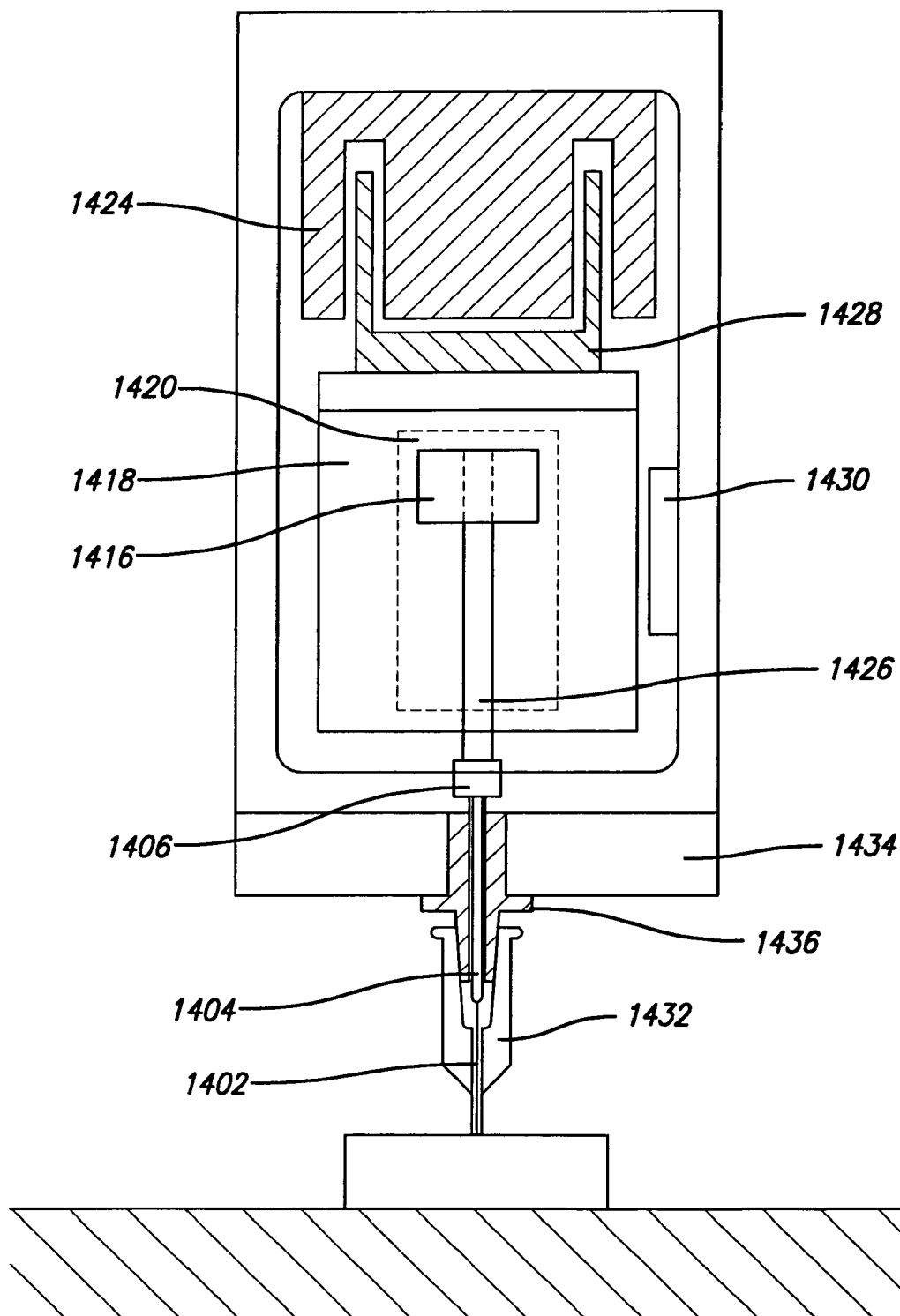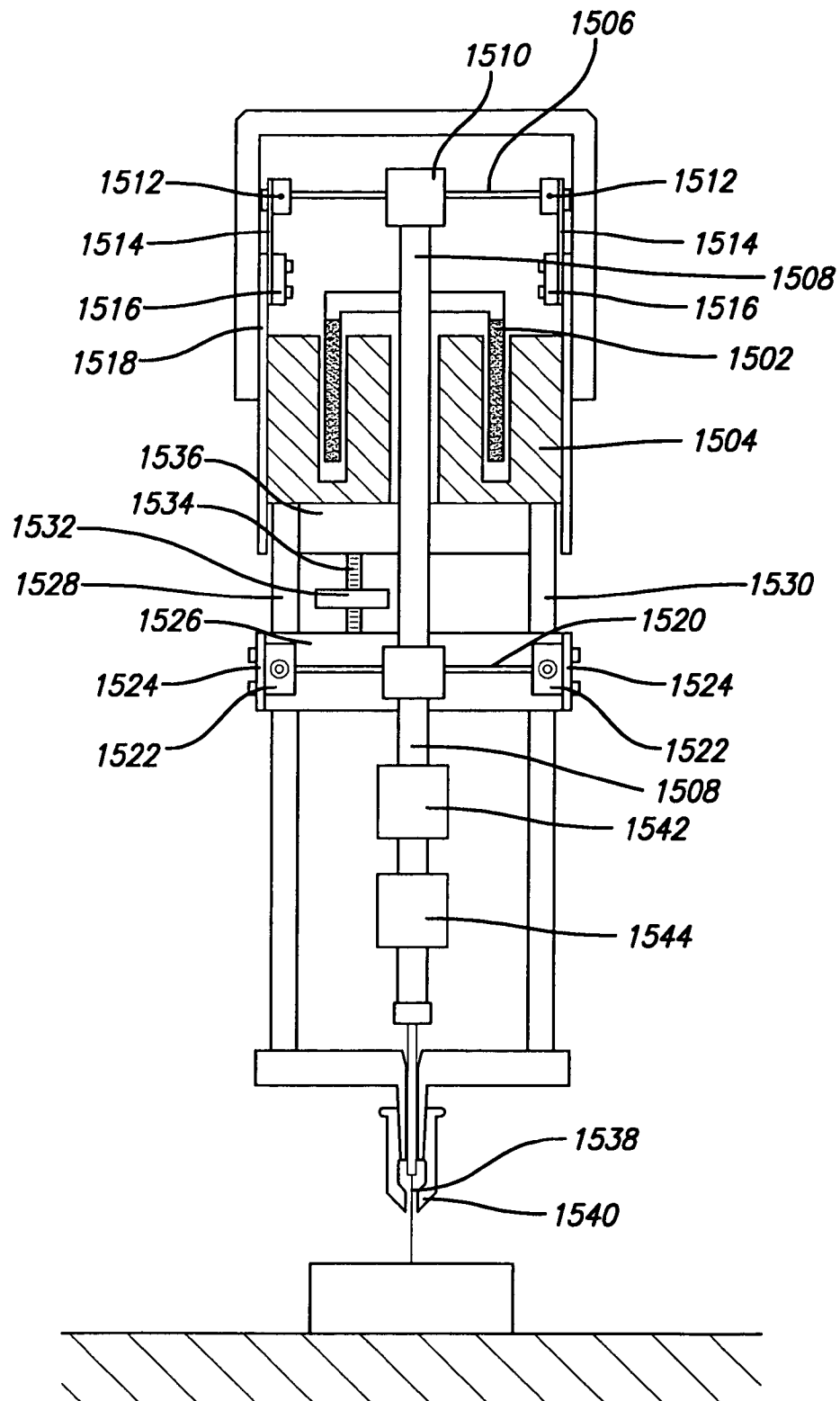
FIG. 15
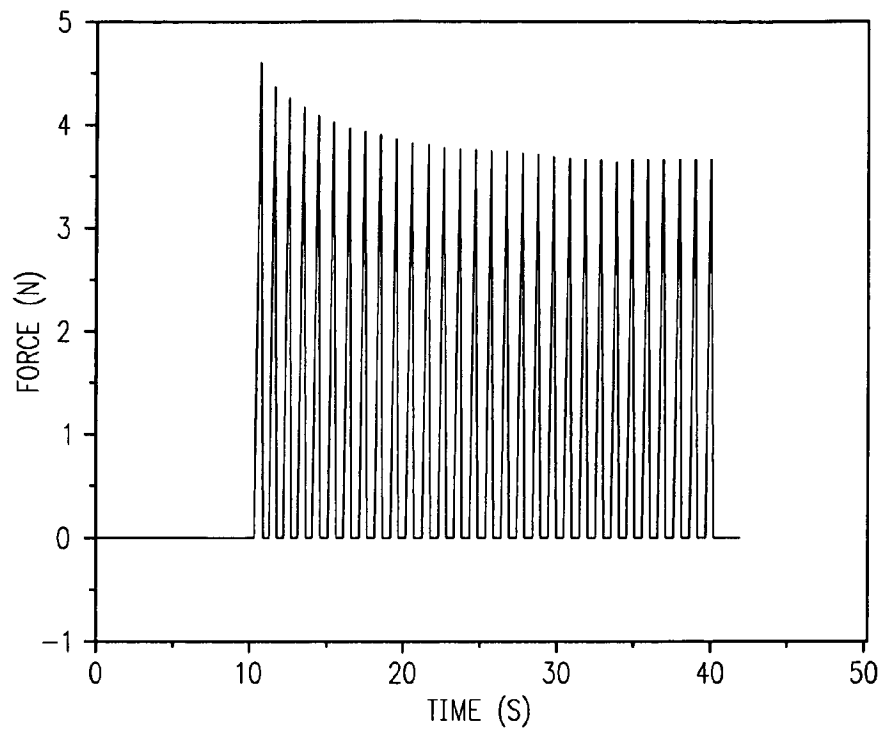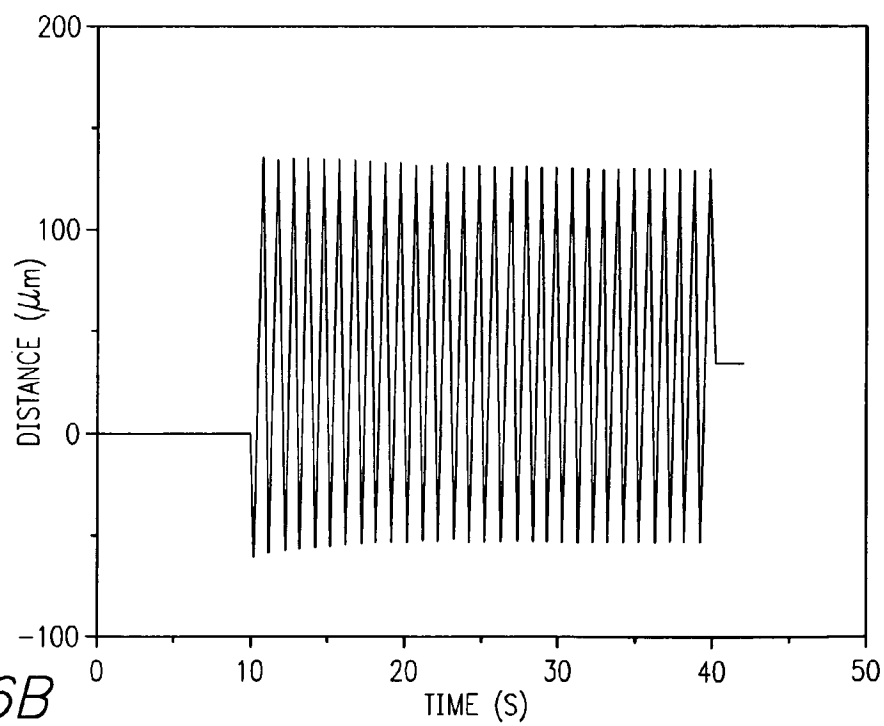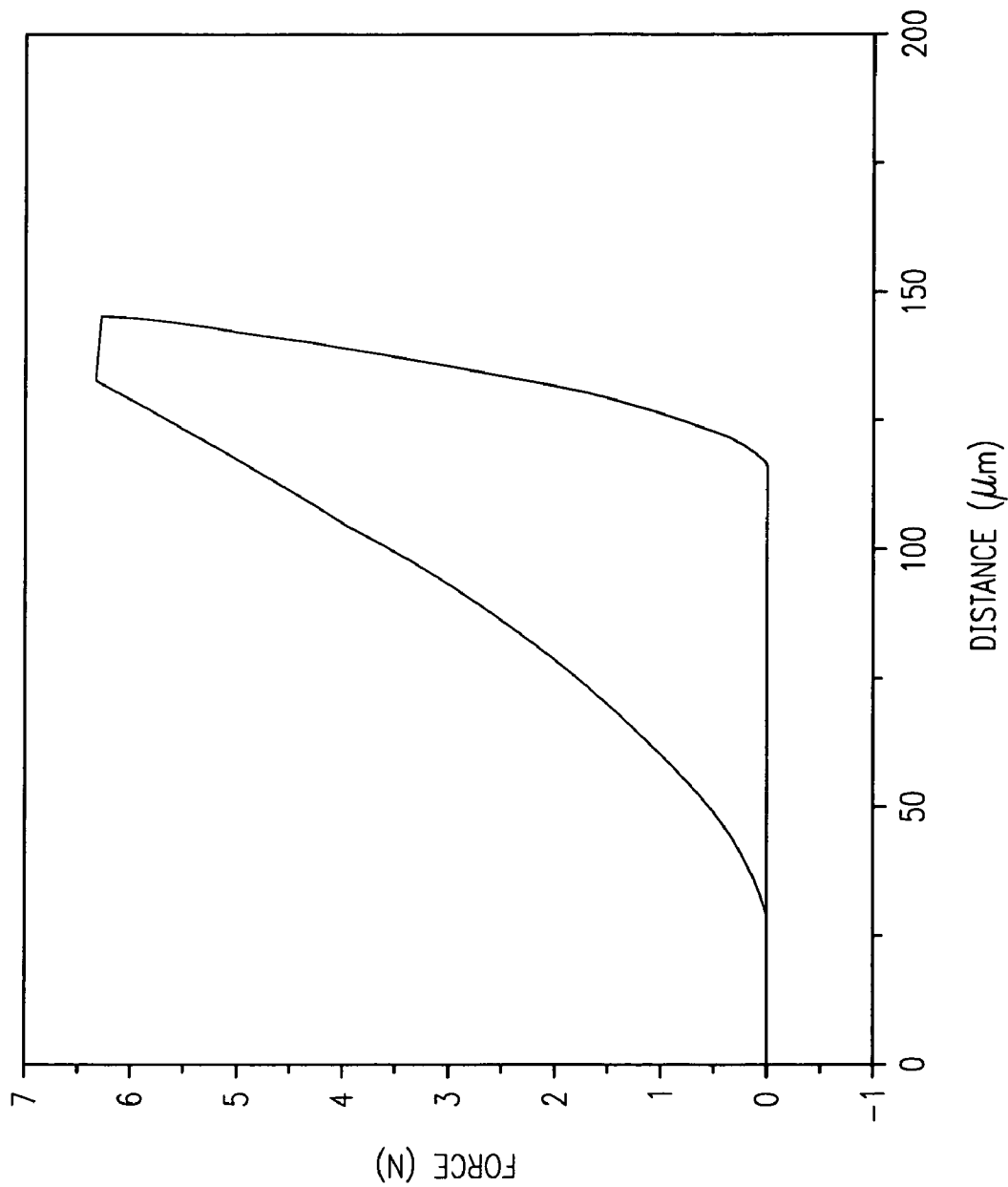
FIG. 16A

FIG. 16B

FIG. 16C
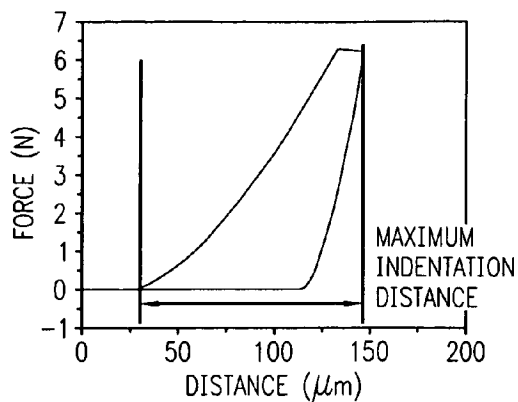
FIG. 17A
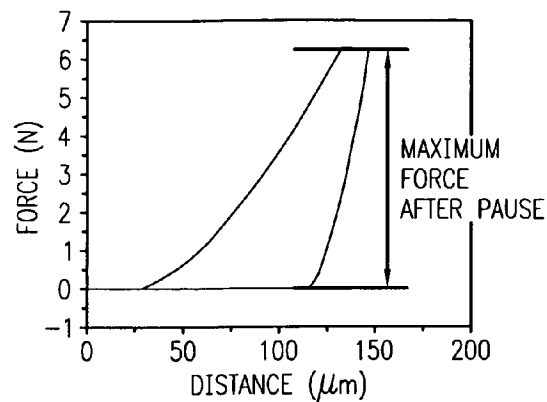
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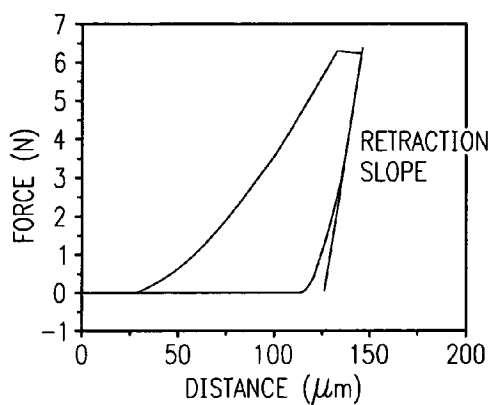
FIG. 17C
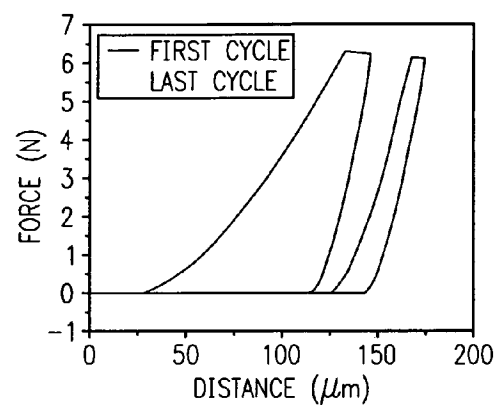
FIG. 17D
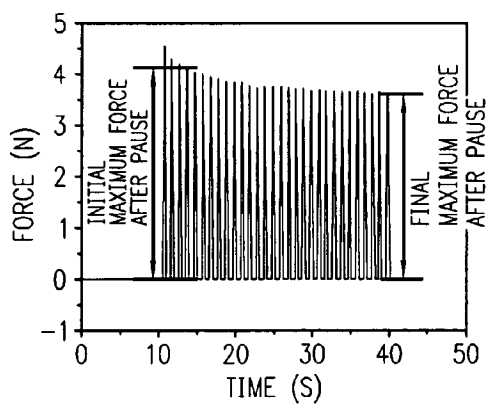
FIG. 17E
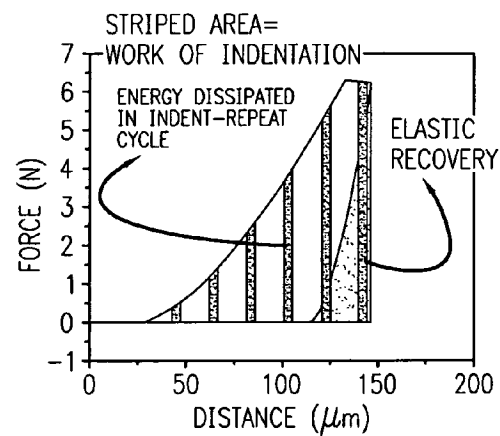
FIG. 17F
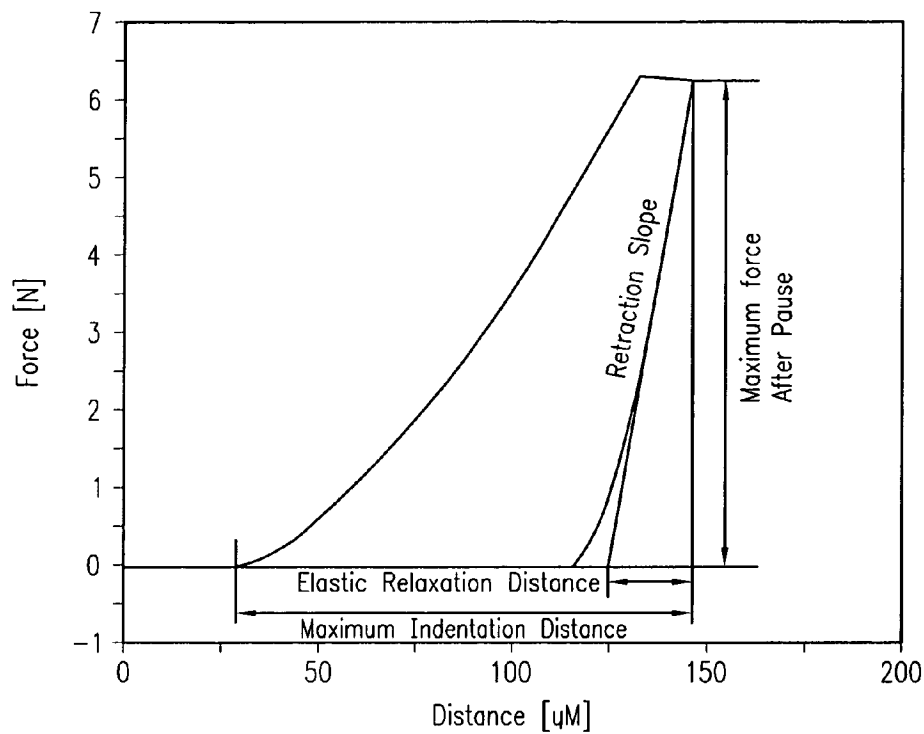
Contact Area A
Retraction Slope S
Maximum Force After Pause $_{max}$
Maximum Indentation Distance h
Contact Indentation Distance h_c
Hardness H
Elastic Modulus E
Poisson Ratio v
Indenter cone half-angle Θ

$$h_c = h - 0.72 \frac{P_{max}}{S}$$

$$A = \pi h_c^2 \tan(\Theta)\sqrt{1 + \tan(\Theta)}$$

$$H = \frac{P_{max}}{A}$$

$$E_{sample} = \frac{1 - v_{sample}^2}{\dfrac{2\sqrt{A}}{\sqrt{\pi}\,S} - \dfrac{1 - v_{indenter}^2}{E_{indenter}}}$$

FIG. 18
PRIOR ART

FIG. 19

FIG. 20

FIG. 21

Data Collection | Data Analysis

(Ff/Fi)/(Df/Di)	Energy Dissipated 2nd final	Elastic Modulus Initial	Elastic Modulus Final/Initial	Hardness Initial	Hardness Final/Initial	Average Adhesion Force/contact Area
1.4792	1.60995	2.73068	1.03014	0.190676	1.02382	4.07E-7

	Retraction Slope	Max Force	Indentation Distance
	0.611985	6.47598	93.2729

Raw Data | Energy Dissipated | Adhesion Force | Elastic Modulus and Hardness

Adhesion force

Plot 0 ◁

[Graph: Y-axis "Amplitude" with values from 0, 1.8-7, 2.8-7, 3.8-7, 4.8-7, 5.8-7, 6.8-7, 7.8-7, 8.8-7, 9.8-7, 1.18-6, 1.28-6, 1.38-6, 1.48-6, 1.58-6; X-axis "Time" with values 0, 1, 2, 3, 4]

FIG. 22

FIG. 23

FIG. 24

METHODS AND INSTRUMENTS FOR MATERIALS TESTING

CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims the benefit of Provisional Patent Application No. 60/921,788, filed Apr. 3, 2007, which is incorporated herein in its entirety.

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

This invention was made with Government support under Grant no. RO1 GM 065354-05 from the National Institutes of Health and Grant no. NCC-1-02037 from NASA. The Government has certain rights in this invention.

FIELD OF THE INVENTION

The invention relates to an apparatus and method for materials testing.

BACKGROUND OF THE INVENTION

Indentation testing to determine the hardness of materials has a long history. Conventional indentation tests include the Brinell hardness test, the Rockwell hardness test, and the Vickers hardness test. The Brinell and Vickers tests involve indenting at a fixed load and then examining the diameter of the indentation. As shown schematically in FIG. 1A, the Rockwell test, which is the most commonly used test, involves measuring the depth of indentation from a fixed load by measuring how far a test probe 102 goes into the material under test 104. This requires a rigid frame 106. It cannot work if there is a soft layer in the mechanical path from the top of the material under test 104 down through the rigid frame 106 and back to the test probe 102 that will deform during indentation (as indicated schematically by the springs 108 in FIG. 1C) because the distance that the test probe 102 goes into the material under test 104 cannot be distinguished from the deflection of the soft layer. A real example of this problem would be attempting to measure the Rockwell hardness of a bone surface exposed during surgery. The soft tissue between the bone and the table on which the body rested would be like the springs 108 shown in FIG. 1C.

The development of very sensitive methods for measuring the depth of indentations such as capacitance sensors, optical beam deflection, laser interferometers or even very sensitive linear variable differential transducers, LVDTs, together with the development of sophisticated techniques for determining mechanical parameters from force vs. distance data only, (ref. W. C. Oliver and G. M. Pharr. Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology. J. Mater. Res. 19 (2004), 3. (review article)), has made possible a new class of indentation machines called nanoindentation testers or nanoindenters. They typically use submicrometer indentations. Nanoindentation testors also use a rigid frame 106 as shown schematically in FIG. 1A to enable accurate measurement of the distance that an indenter goes into the sample at a fixed load for macroindentation tests or variable loads for nanoindentation tests. Again, a substantial soft layer under the sample as shown in FIG. 1C would prevent accurate nanoindentation testing.

This solution to the problem of soft layers has been previously implemented, for example, in U.S. Pat. No. 1,770,045,

with a durometer as shown in FIG. 2A. In this case a rigid frame is not needed because the base of the durometer 202 rests directly on the material under test 204 and indentations of the test probe 206 (sometimes called the foot) into the material are measured relative to the position of the base of the durometer 202. However, durometer indentation 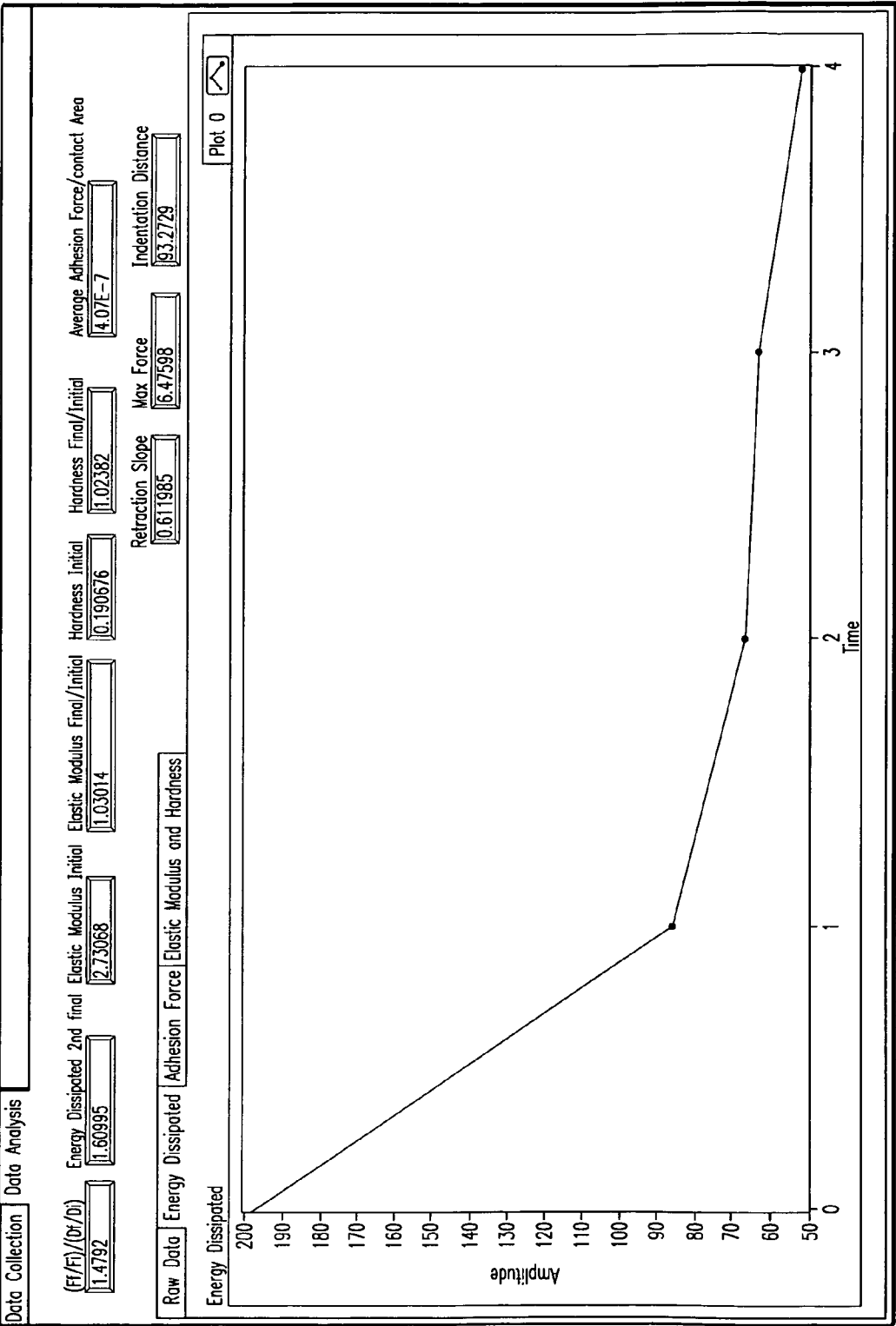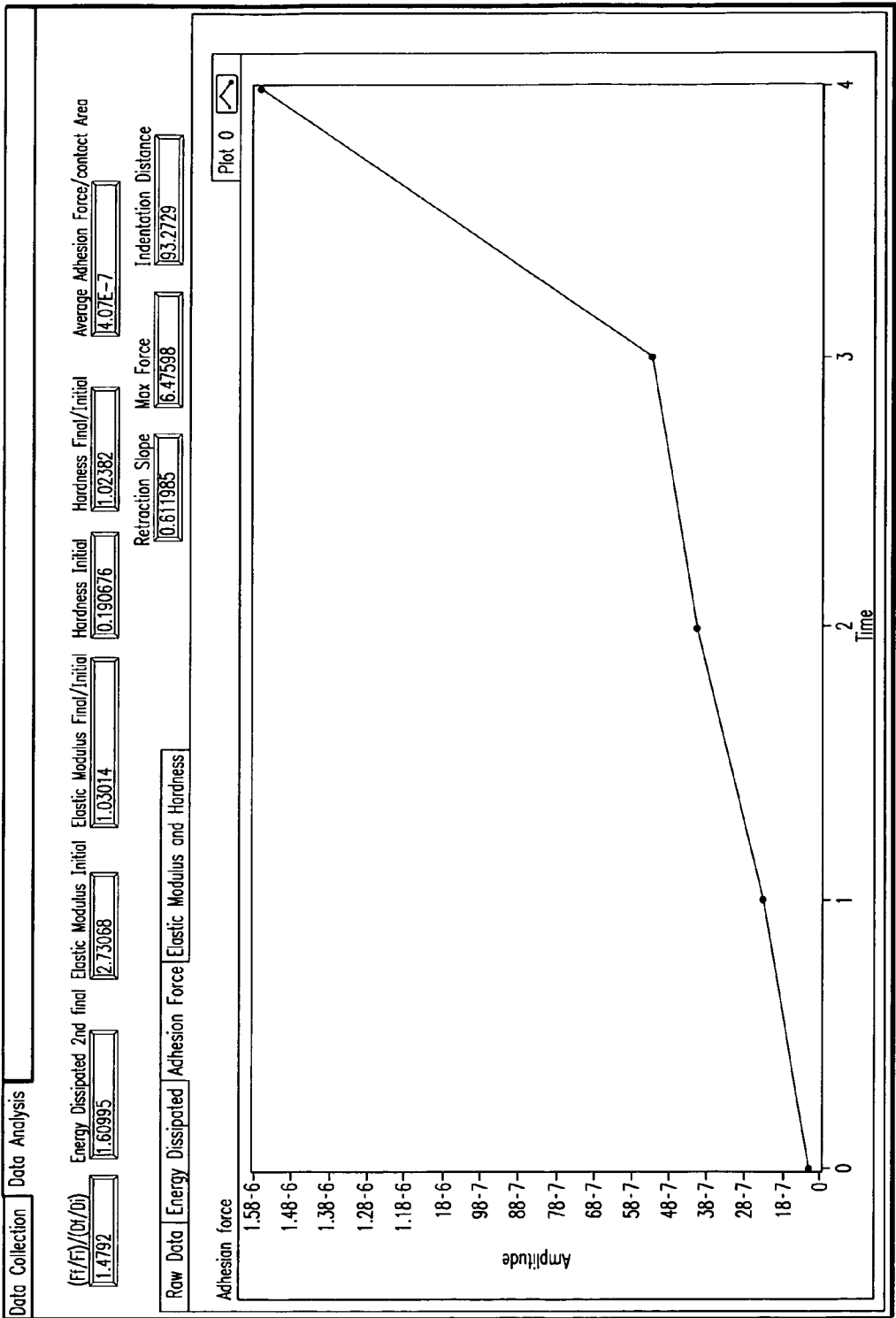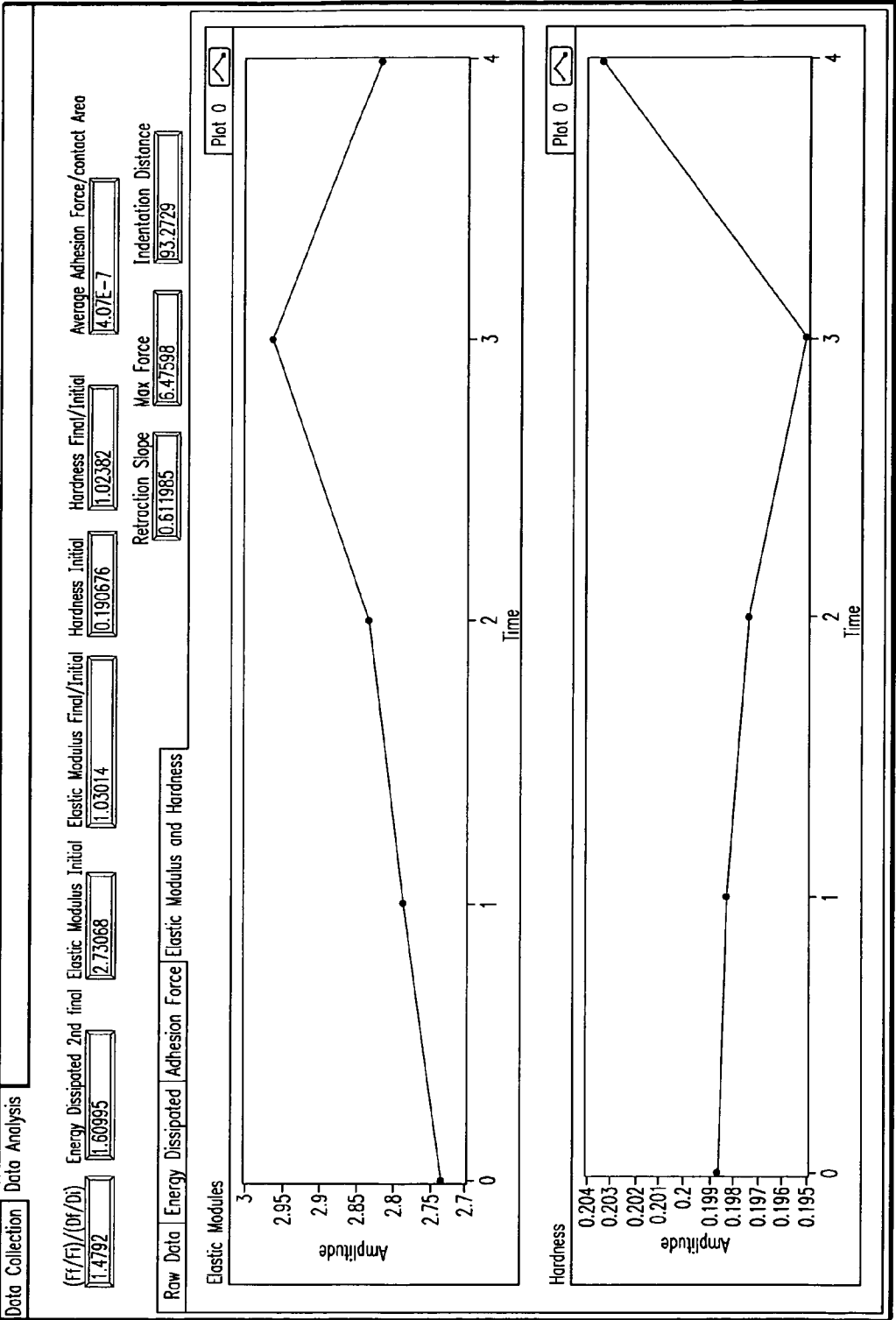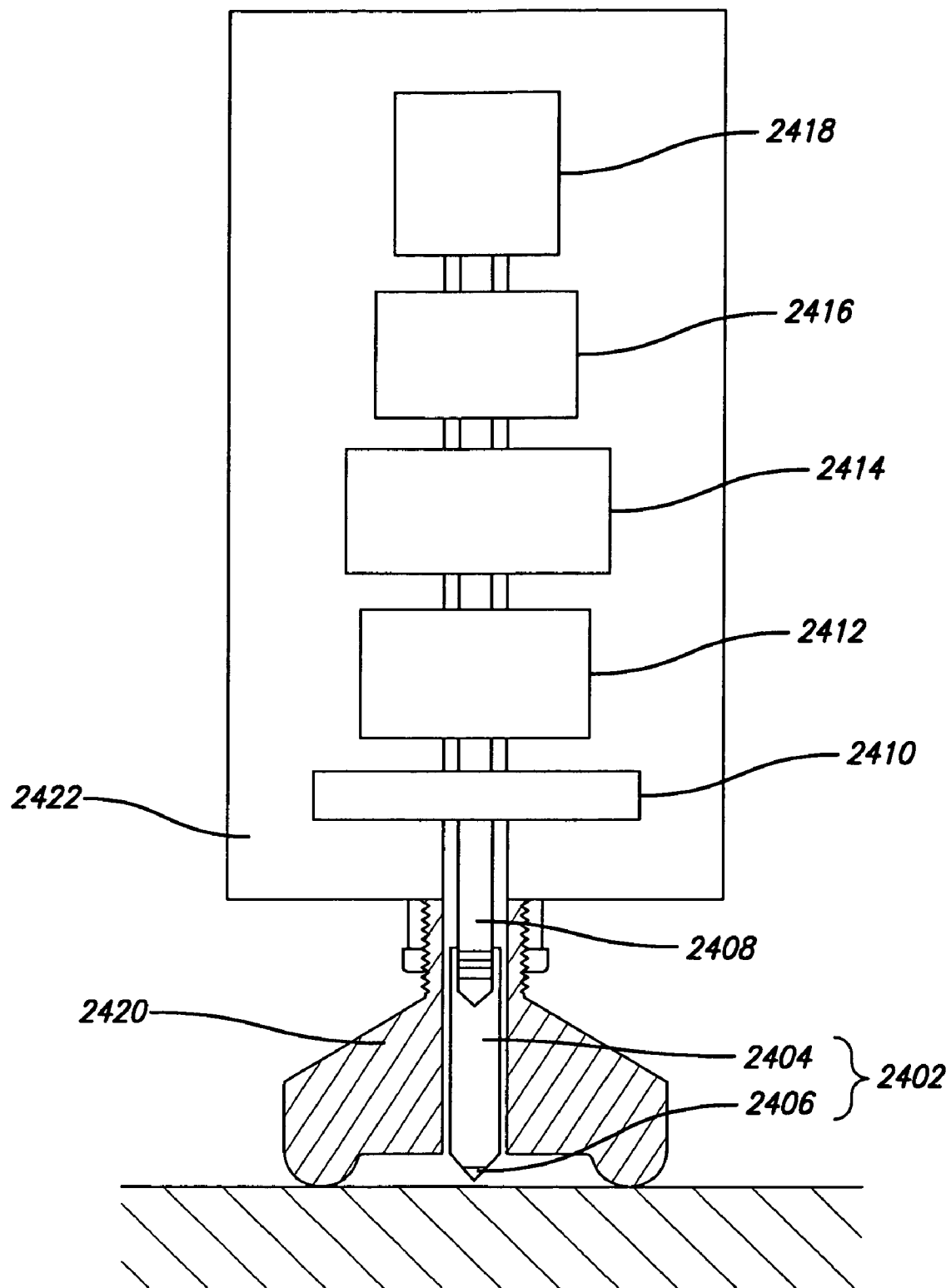measurements only characterize the material with a hardness number. Attempts have been made to relate hardness measurements taken with a durometer to the elastic modulus of the material. However, no accurate, widely accepted model is available. This is in part due to the difficulties in theoretical analysis arising from the complex indenter geometry, and the inability to correct for time-dependent effects because of a lack of control of the loading rate with the durometer [Briscoe, B. J. and Sebastian, K. S. An analysis of the durometer indentation. Rubber Chemistry and Technology 66 (5): 827-836 1993)].

Other prior art portable hardness testers also exist. In particular there are many rebound testers such as the TH130 and TH150 pocket-size hardness tester from Corvib and many ultrasonic hardness testers such as the High Resolution SH-21 Portable Hardness Tester from Micro Photonics Inc. Here too, however, to the best of our knowledge there exists no portable tester that measures more material properties beyond just hardness.

One approach to indentation measurement on soft samples is to use, as a distance reference, the upper surface of the sample as is found in the instrument outlined in U.S. Pat. No. 6,142,010. In spite of this improvement, this instrument is limited in that it is solely designed for measuring hardness and relies on an external mechanical frame (as opposed to a reference probe) to maintain a rigid mechanical path between the sample and the distance measurement. The upper surface of the sample is used for a differential measurement of the indentation depth in the CSM Indentation Testers, which can measure more that just hardness. Here again, however, a rigid frame is present.

Atomic Force Microscopes (AFMs) can rest on the surface of the material under test and could, in principle at least, be used for indentation tests [C. A. J. Putman, H. G. Hansma, H. E. Gaub, and P. K. Hansma, Langmuir 8, 3014 (1992)]. An example of indentation tests on bone with the AFM is James B. Thompson et al., Nature 414, 774, 13 Dec. 2001, though this was done with a prototype AFM that was not capable of resting on the surface of the material under test.

One AFM company, Asylum Research, has also produced a nanoindenter, the MFP-3D NanoIndenter™ for Quantitative Surface Characterization. This instrument eliminates the problem of angular motions of cantilevers and goes to higher forces, up to 14 milliNewtons. It consists of a new NPS™ Nanopositioning sensor for their MFP-3D™ Stand Alone Atomic Force Microscope. The sample is held rigidly to the MFP-3D scanner through specialized sample mounts. Thus it is not designed to rest on the surface of the sample as for the present invention.

Other publications dealing with prior art systems include: U.S. Pat. No. 5,450,745; U.S. Patent Publication Nos. 2002/0170360 and 2005/0262685; "Micro Hardness Tester (MHT) for fracture toughness determination of brittle materials", No. 8, July 1998; CSM Indentation Testers, four page brochure; and "ASTM Proposed Instrumented Indentation Testing Standard", pages 1-4, October 2003.

Thus, while there have been portable hardness testing devices and devices that measure parameters other than hardness, we are aware of no prior device that combine the ability to be portable with the ability to measure a wide variety of parameters based on indentation of a probe into a sample.

BRIEF SUMMARY OF THE INVENTION

The present invention overcomes the foregoing drawbacks in providing improvements in the technology for measuring material properties of materials such as bone in a living person, teeth, the leg bones of race horses, the wing of an aircraft, the surface of a part during manufacturing or assembly and other structures that are not easily tested in conventional mechanical testers.

The invention is designed to measure more parameters than just hardness. This is important for many applications such as predicting bone fracture resistance or monitoring fatigue damage in airplane wings where measurements of hardness alone are not sufficient. Thus, compared with the devices referred to above in the BACKGROUND OF THE INVENTION section, the invention extends the capabilities of previous instruments for measuring the material properties of materials under test by making it possible to measure more material properties than just hardness with a portable instrument. Moreover, the instrument can be portable and hand held. At the heart of the invention is a measurement head that contains a reference probe that rests substantially on the surface of the material under test and provides a reference for measuring the distance that a test probe indents the material under test. The invention can, optionally, measure complete force vs. distance curves during one or multiple indentation cycles where the force is the force that the invented instrument supplies during the indentation cycle(s).

More particularly, in a departure from prior devices, we provide a device and method for characterizing a material using a test probe and constructed for insertion into the material and a reference probe aligned with the test probe in a housing. The housing is hand held or placed so that the reference probe contacts the surface of the material under pressure, applied either by hand or by the weight of the housing, causing the test probe to indent the material while maintaining the reference probe substantially under said pressure. This allows the evaluation of one or more properties of the material related to indentation of the material by the probe.

Referring again to FIGS. **1A**-**1D**, the invention replaces the rigid frame **106** with a reference probe **110** that rests directly on the surface of the sample. Now, as will be further detailed below, the relevant mechanical path will be from the material up through the reference probe **110** and back down to the test probe **102**.

The invention increases the capability of the durometer by adding a measurement head containing electronic actuators to generate forces and/or displacements as well as sensors for load and displacement that are coupled to a computerized data generation, collection and analysis system to get many parameters beyond just hardness. Compared with other instrumented indentation systems capable of measuring properties beyond hardness, our invention greatly increases the ability to test samples with complex geometries or in locations where attachment of a sample to a rigid sample holder is impossible.

The invention is also distinct from the above described AFMs in that the test probes of the invention are not mounted on cantilevers as for the AFMs. Thus there is not the problem of angular motions of cantilevers. Also, the preferred embodiments of the present invention typically go to much larger forces, several Newtons, compared to the microNewtons, nanoNewtons or below as typical of AFMs. This is an advantage for testing real materials without special surface preparation because the probed volume is large enough to be insen-

sitive to thin surface layers of, for example, the water that covers most materials in ambient environments, and surface topography.

An additional feature of the invention is an optional wireless connection between a portable measurement head, which contains the mechanical components necessary for the measurements together with some electronics, and a base station, which contains electronics including, optionally, a computer. The base station can both supply instructions for the measurements and acquire data from the measurements.

Another additional feature of the invention is the optional ability to hand-hold the measurement head. This increases the ease and speed with which measurements can be made on complex structures such as airplane landing gear or a race horse's leg. The combination of wireless operation and a hand held measurement head is particularly useful for measurements in the field: outside a testing lab.

Still another feature enabling a compact hand-holdable instrument with the extensive capabilities of the invention is the use of opposing flexures with a linear translation stage that facilitate incorporation of a voice coil actuator.

BRIEF DESCRIPTION OF THE DRAWINGS

For a more complete understanding of the present invention, reference is now made to the following descriptions taken in conjunction with the accompanying drawing, in which:

FIGS. **1A**-**1D** are schematic drawings illustrating the advances in the present invention over the prior art instruments that rely on a rigid frame for measuring the depth of indentation;

FIGS. **2A** and **2B** are schematic drawings illustrating the advances in the present invention over the prior art instruments called durometers;

FIG. **3** is a cross-sectional view of the measurement head of this invention in accordance with one embodiment;

FIG. **4** is a cross-sectional view of the measurement head of this invention in accordance with one embodiment;

FIGS. **5A** and **5B** depict a portable embodiment of the instrument with wireless connection between a portable measurement head of this invention, which contains the mechanical components necessary for the measurements together with some electronics, and a base station, which contains electronics including, optionally, a computer;

FIGS. **6A**-**6C** depict the flexures used in the embodiments of FIGS. **3** and **4**;

FIG. **7** depicts the finite element analysis of the flexure shown in FIG. **6**;

FIGS. **8A**-**8D** depict an alternate design for the flexures used in the embodiments of FIGS. **3** and **4**. This flexure has lower stiffness than the one depicted in FIG. **6**;

FIGS. **9A**-**9E** depict various reference probes that can be used with the measurement head of this invention shown in FIG. **3**;

FIG. **10** depicts various test probes for the invention;

FIG. **11** depicts probe assemblies, which are combinations of test probes with reference probes, that can be used with the measurement heads of this invention show in FIGS. **4**, **13**, **14** and **15**, showing also various ways to attach a test probe to the shaft of the measurement head of this invention and various ways to inhibit buckling of the test probe;

FIG. **12** depicts various support stands for the measurement head used in the invention;

FIG. **13** is a view of the measurement head of this invention in accordance with one embodiment that contains mecha-

nisms for both a coarse and fine adjustment of the relative position of a test probe and a reference probe;

FIGS. **14A** and **14B** are views of the measurement head of this invention in accordance with two embodiments that use a linear stage rather than flexures;

FIG. **15** is a view of the measurement head of this invention in accordance with one embodiment that uses wire flexures rather than the disk flexures of FIGS. **3** and **4** or the plastic flexures of Clark Synthesis force generator shown in FIG. **13**;

FIGS. **16A-16C** show three types of data graphs that can be generated with this invention;

FIGS. **17A-17F** show various parameters that can be extracted from data graphs generated with this invention;

FIG. **18** shows prior art equations [W. C. Oliver and G. M. Pharr. Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology. J. Mater. Res. 19 (2004), 3.] for calculating Hardness and Elastic Modulus;

FIG. **19** shows a screenshot of the user interface of the Labview program used in the currently preferred embodiment to control the invention;

FIG. **20** shows a screenshot of the automated data analysis interface of the Labview program used in the currently preferred embodiment;

FIG. **21** shows a screenshot of the automated data analysis curve of Energy Dissipated as a function of time from the Labview program used in the currently preferred embodiment;

FIG. **22** shows a screenshot of the automated data analysis curve of Adhesion force as a function of time from the Labview program used in the currently preferred embodiment;

FIG. **23** shows a screenshot of the automated data analysis curves of Elastic Modulus and Hardness as a function of time from the Labview program used in the currently preferred embodiment; and

FIG. **24** depicts a generalized measurement head used in the invention.

DETAILED DESCRIPTION OF THE INVENTION

The essential feature of the invention is a probe that is inserted into a material under test a distance that is measured relative to a reference probe, which rests substantially on the surface of the material under test. In a preferred embodiment, the probe consists of a steel shaft tipped with a sharpened diamond. It slips inside a three footed reference probe with feet on a circle of diameter approximately 1.5 inches. A typical penetration depth is 0.05 mm.

The method is particularly suited to evaluating one or more properties of a living human bone of a subject, the test probe being inserted through the periosteum and/or soft tissue on the bone so that the test probe contacts the subject's bone. Such an evaluation is described in our previous work on developing a bone diagnostic Instrument, filed in U.S. patent application Ser. No. 11/417,494 filed May 3, 2006 titled Methods and Instruments for Assessing Bone Fracture Risk, the disclosure of which is incorporated herein by reference.

In one class of embodiments, the probe and reference probe are connected to a measurement head. This measurement head can be connected to a base station either with wires or with a wireless connection. This measurement head can be held on the material under test with a stand or it can be hand held or it can be hand held with the help of an optional support. This optional support can, for example, serve to hold the measurement head at a fixed angle relative to the surface of the material under test during the test process.

The test process consists of one or more indentation cycles during which the force applied to the probe and the distance that the probe is inserted into the material under test are measured. These measurements can be analyzed to give material parameters such as Elastic Modulus, Hardness, Adhesion (that is the maximum force needed to pull the probe out of the material), Elastic Energy Dissipation, Plastic Energy Dissipation, Total Energy Dissipation, Maximum distance of insertion, and Maximum Force during the cycle. All of these parameters can be measured as a function of time through a series of cycles. For example, we sometimes measure bone parameters over a series of 80 cycles at 3.5 Hz. Additional useful parameters are the ratios of the final to initial values of the individual parameters: for example, the ratio of the Plastic Energy Dissipation on the final cycle to the Plastic Energy Dissipation on the first cycle. It has turned out that the ratio of the final value of Hardness to the initial value of the Hardness on bone is correlated with resistance to bone fracture.

FIG. **3** is a detailed drawing of the measurement head of a currently preferred embodiment of our invention. A test probe **302** consisting of a shaft **304** and a sharp tip (often a diamond) **306** is attached to a shaft **308** that connects to the core **310** of an LVDT **312** (for example Measurement Specialties MHR 025). This in turn connects to a load cell **314** (for example Futek LSB 200) and then, with a shaft **315**, to a force generator **316** consisting of two flexures of novel design **318**, (which will be described in more detail in FIGS. **6**, **7** and **8**) together with a voice coil actuator (a modified version of BEI Kimco Magnetics LA16-27-000A) which consists of a moving coil **320** in a magnetic field assembly **322**. The flexures **318** are attached with screws **323**. This force generator **316** is anchored in an inner shell **324** that is capped by the flexures **318**. The force generator **316** is held in an outer shell **326**. The outer shell is connected to a nose piece **328**, which supports the LVDT body **312**. The position of the LVDT body **312** can be adjusted to zero or otherwise adjust the signal from the LVDT **310**, **312** with a fine screw **330** and is locked into place with set screws **332**. The nose piece **328** also rigidly supports a reference probe **334** that rests on the surface of the sample under test **336**. The measurement head can be hand held during the test, which has the advantage that it can be forced against the sample under test with greater force than its own weight. In general the largest force that can be applied by the force generator **316** to the sample under test **336** must be less than the force with which the measurement head is pressed against the surface. Otherwise the measurement head will lift off the sample under test **336**. This greater force with hand held operation allows greater maximum force during the measurement of force vs. distance curves. Hand held operation also allows measurements on surfaces that are not substantially horizontal and measured from above. Alternately, elastic elements such as springs or rubber tubes or bungee cords can used to hold the measurement head against the sample under test.

The electrical signals to actuate the force generator **316** as well as the force signal from the load cell **314** and the distance signal from the LVDT **310**, **312** pass through an electrical connector **338** (AMP **28** pin connector). An optional adjustment of the initial position of the test probe **302** relative to the reference probe **334** can be made with an optional spring **340** that is pulled with an optional screw **342** that is threaded through a cap **344**. The bandwidth of both the LVDT **310,312** and the load cell **314** and both their amplifiers and the data acquisition system is 1 kHz or above. Thus the instrument can be operated to obtain complete force vs. distance curves in cycle times as fast as 0.1 second. For maximum resolution, cycle times as fast as 1 second are more typical. For maximum

speed, cycle times as fast as 0.01 second have been used, but the force vs. distance curve is not accurately captured. Fast cycle times can, however, be used to test for damage during cyclic loading with, optionally, slower, more accurate curves taken before and after the fast cyclic loading.

FIG. **4** is a detailed drawing of another version of the measurement head of a currently preferred embodiment of our invention. This version is designed to be used as Bone Diagnostic Instrument (Methods and Instruments for Assessing Bone Fracture Risk U.S. patent application Ser. No. 11,417,494) for which soft tissue overlying the bone must be penetrated in order to measure the properties of the underlying bone. In this version the test probe **402** is a sharpened steel rod of diameter 0.015" held in a mounting pin **404** which attaches magnetically to a permanent magnet **406** that is attached to a shaft **408** that connects to the core **410** of an LVDT **412** (for example Measurement Specialties MHR 025). This in turn connects to a load cell **414** (for example the Futek LSB 200 or the Sensotec Model 34 precision miniature load cell) and then to a force generator **416** consisting of two flexures of novel design **418**, which will be described in more detail in FIGS. **6**, **7** and **8**) together with a voice coil actuator (a modified version of BEI Kimco Magnetics LA16-27-000A) which consists of a moving coil **420** in a magnetic field assembly **422**. This force generator **416** is anchored in an inner shell **424** that is capped by the flexures **418**. The force generator **416** is held in an outer shell **426**. The outer shell is connected to a nose piece **428**, which supports the LVDT body **412**. The position of the LVDT body **412** can be adjusted to zero or otherwise adjust the signal from the LVDT **410**, **412** with a fine screw **430** and is locked into place with set screws **432**. The nose piece **428** also rigidly supports a reference probe **434** that rests on the surface of the sample under test **336**. In this version the reference probe **434** consists of small diameter stainless steel tubing **436** held in a brass body **438** that is threaded into the nose piece **428** and held rigidly in position with a knurled locking nut **440**

The electrical signals to actuate the force generator **416** as well as the force signal from the load cell **414** and the distance signal from the LVDT **410**, **412** pass through an electrical connector **442** (AMP **28** pin connector). An optional adjustment of the initial position of the test probe **402** relative to the reference probe **434** can be made with an optional spring **444** that is pulled with an optional screw **446** that is threaded through a cap **448**.

FIGS. **5A** and **5B** shows how the currently preferred embodiments of our invention can be made wireless. The measurement head **503** (shown in more detail in FIGS. **3** and **4**) is combined with a wireless adapter/power pack module **500** for the instrument. This module **500** has many functions including: supplying power for the instrument, amplifying and conditioning the signals from the transducers, the transmission of data, the reception of input signals, and the amplification of input signals.

The transmission of the data starts at the connector **501**, where the interface with the instrument is located. The signal for the distance sensor (from the instrument) is sent through the connector **501** to the PC board with distance sensor amplifier and signal conditioner **504**. Here the signal is amplified and then sent to the wireless module **508**. The data is then sent to the partner computer(s) **513** (FIG. **5B**) with electromagnetic radiation **509**. The signal from the load cell (from the instrument) goes through the connector **501** to the PC board with load cell amplifier and signal conditioner **502**. Here the signal is amplified and conditioned and then sent to the wireless module **508** where it is transmitted to the partner computer(s) **513** with electromagnetic radiation **509**.

Input signals are received via electromagnetic radiation **509** by the wireless module **508**. The signals are then transmitted to and amplified by the PC board with amplifier **506** that drives the force generator **416**. The amplified signal is then sent to the force generator **416** in the instrument via the connector **501**.

The whole system, including the instrument, may be powered from the rechargeable battery **510**. The battery itself has an energy port **511** where external power can be introduced into the system to either recharge the battery or power the wireless module externally. The battery **510** may also be wired such that it is easily removable or replaceable. Batteries such as found in small hand tools such as cordless drills are suitable. For the force generator **416** in the currently preferred embodiment, we typically use average currents of less than 1A at voltages of a few Volts for times of order 20s thus requiring of order 0.0056 Ah per test. This can easily be supplied by the type of NiMH rechargeable batteries used in cordless drills, which can supply 3 Ah, enough for over 500 tests.

The wireless adapter/power pack module **500** can (optionally) also contain a keypad **512** to set test parameters and access selected test data and analysis on a display screen **514**. For wireless use, the switch **516** can be added to the measurement head **503** (shown in more detail in FIGS. **3** and **4**) to trigger test cycles conveniently.

The measurement head **503** is connected to the wireless adapter/power pack module **500** with cable **518**. As shown in FIG. **5A**, the cable can be external, joining the measurement head **503** to the wireless adapter/power pack module **500** that is mounted on top of the measurement head. This permits the removal of the wireless adapter/power pack module **500** if it is desired to have the measurement head connected directly to control electronics and a computer. If the unit is designed only for wireless use, the cable can, of course, be internal. Alternately the wireless adapter/power pack module **500** can be separate from the measurement head **503** as shown in FIG. **5B**. This has the advantage of making the hand held part lighter and the wireless adapter/power pack module **500** more capable in terms of battery capacity, data processing and data storage.

FIGS. **6A-6C** show the circular flexures used in the currently preferred embodiment of the invention. The flexures are included to guide the motion of the force generator (for example **416**, FIG. **4**) and ensure that there is no substantial off-axis motion. The flexures design consists of a large, horizontal, thin inner membrane **602** connected to a outer, thin, horizontal membrane **604** through a vertical ring **606**. The design of the flexures was improved through the use of finite element analysis. FIG. **7** shows the results of simulating the deformation of one of the flexures under an axial load.

The softness of the circular flexure may be increased by cutting out radial sections **802** of the flexure, as shown in FIGS. **8A-8C**. Further softening could be achieved by cutting away more of the circular flexure, for example, leaving only thin radial strips like spokes on a wheel.

FIGS. **9A-9E** show various reference probes that can be used in place of the reference probe **334** shown in FIG. **3**. **9A** has three rounded feet **900** to minimize marring of the material under test. **9B** has three pointed feet **902** to minimize lateral slipping. **9C** has three adjustable feet **904** that contain permanent magnets **906** to minimize slipping and marring of magnetic surfaces such as steel. These magnets **906** could also be electromagnets or mechanically switchable magnets as used in magnetic bases (see, for example, **1228** in FIG. **12**). **9D** shows one of many possible variants of fixed **908** and adjustable feet **910**. The motion of the adjustable foot **910** is

demagnified at the location of the test probe 912 to give more precise positioning. 9E shows a reference probe that is suitable for use with a standard diamond indenter 914. As an example, a Rockwell Diamond Indenter with a Versitron shank 914 is shown, but, of course many others could be used with a suitably designed reference probe.

FIG. 10 shows different possibilities for the tips of test probes. Test probe 1000 is patterned on the diamond indenter used in Knoop hardness testing. It has a pyramid-shaped diamond 1002 with apical angles of 130° and about 170°, mounted on a tungsten carbide shank 1004. Test probe 1006, has a diamond 1008 in the shape of a square-based pyramid whose opposite sides meet at the apex at an angle of 136° as used in Vickers hardness testing of metals and ceramics, mounted on a ceramic shaft 1010. Test probe 1012 is a disk that can be rotated for measuring friction, $\phi=0$, or viscosity of tissue near a bone surface, at $\phi=0$ or $\varnothing>0$ as in conventional viscosity measurements. Test probe 1014 is wedge shaped and is used for assessing the fracture resistance of materials. Test probe 1016, designed for testing the material properties of bone and teeth, has a cone at its end. In a preferred embodiment $\theta=90$ and the test probe is tool steel. In other embodiments the test probe can have angles $\theta=70$ and 50 and can have a tip 1018 of a different material, such as diamond. Test probe 1020 is patterned after the indenters used in some Rockwell and Brinell hardness testing, and has a half sphere of tungsten carbide 1022 bonded to a steel shank 1024. Test probe 1026 is a tube that can be rotated for measuring friction on the surface of a material. Test probe 1028 is a screw that can test bone by measuring the torque necessary to screw it into the bone. These are only intended as representative examples. Many other geometries and test probe materials could be used.

FIG. 11 shows details of three probe assemblies (test probes in reference probes) in the top row. In the first example, the test probe 1102 is held in a mounting pin 1104 that is a 1/16" diameter steel rod with a hole in the end into which the test probe is glued or soldered. The reference probe 1106 is composed of sharpened, small diameter tubing 1108 joined to a threaded body 1110. In the second example, the test probe 1112 is shorter, but, when mounted on a longer mounting pin 1114, gives the same overall length of mounted test probe as the previous example. In this case the reference probe is a hypodermic syringe needle 1116 that is removably mounted in a threaded Luer adaptor 1118. In the third example, the test probe 1120 is mounted in a mounting pin 1122. Here the reference probe 1124 has no tubing projecting from the end, but is suitable for use when the material under test is not covered with a layer that must be penetrated (as in the case of skin covering bone).

FIG. 11 also shows, in the bottom row, some details of an alternate to the magnet shown as 406 in FIG. 4 for holding mounted test probes. The collet 1126 holds the mounting pin 1128 for the test probe 1130. This collet 1126 is attached to a shaft 1132, shown as 408 in FIG. 4. The collet 1134 holds the test probe 1136 directly. The tube 1138, which can optionally be attached to the test probe 1136, functions to minimize buckling of the test probe 1136. The collet 1140 holds a tube 1142 in which the test probe 1144 is mounted.

Finally, the magnet 1146 holds the mount 1148 for the test probe 1150. This test probe and all the test probes in FIG. 11 can have many tip shapes, as shown in FIG. 10.

FIG. 12 shows various support stands for the instrument. The instrument can be hand held, resting on the three feet of the reference probe 334 that rest on the surface of the sample under test 336 shown in FIG. 3. More reference probes for hand held use were shown in FIGS. 9A-9E. For some appli-

cations, however, it is useful to supplement or eliminate the hand holding the instrument. The goal is to stabilize the instrument more than is possible when just hand held. As some examples, the measurement head 1202 is attached with a removable mount 1204 (for example, a 1/4-20 screw mount such as used for cameras) to a rail 1206 which is, in turn held in a guide block 1208 (for example, the Miniature Corrosion-Resistant Versa-Mount Guide Blocks and Rails form Mc Master Carr can be used) which is, in turn, attached with a removable mount 1210 (for example, a 1/4-20 screw mount such as used for cameras can be used here also) to a support arm 1212 attached to a base 1214. This support stand allows the measurement head 1202 to move freely up and down while being constrained laterally and held vertically.

The measurement head 1216 is also mounted via a removable mount 1218 to a rail 1220 which is, in turn held in a guide block 1222 (for example, the Miniature Corrosion-Resistant Versa-Mount Guide Blocks and Rails from Mc Master Carr can be used) which is, in turn, attached with a removable mount 1224 (for example, a 1/4-20 screw mount such as used for cameras can be used here also) to an adjustable arm 1226 attached to a magnetic base 1228.

The measurement head 1230 is permanently mounted by attachments at each end to a rail 1232, which is, in turn, mounted to an articulating arm system 1234 (such as the FlexArm available from Midwest Specialties Inc.). The probe assembly 1236 is shown schematically penetrating soft tissue of a leg 1238 to reach the tibia 1240. The leg is held in a modified V block 1242 to stabilize it during the measurements.

The measurement head 1244 is held in a microscope stand 1246. The material under test is in a fluid cell 1248 that is filled with fluid 1250. One of the advantages of all the embodiments shown is that it is easy to work with samples under aqueous buffers to, for example, simulate physiological conditions. It is also easy to put in a heating stage or hot plate under the fluid cell 1248 or under a material under test that is not in a fluid cell because there is no rigid frame that limits the space below the measurement head. Though FIG. 12 shows a particular microscope stand 1246, a wide variety of microscope stands are available for mounting stereo microscopes including ones for operating rooms that roll on the floor and allow the surgeon to see parts of a patient's body on the operating table. This type of rolling microscope stand could hold the measurement head 1244 for testing the bone or teeth of a patient on a table. The core assemble is mounted in a disk of the correct diameter for the particular microscope stand (typically about 3" in diameter). This mounting in the disk can be rigid (as shown) or via a rail and guide block system as shown in the other support stands. Conversely the other support stands can be used without a rail and guide block system. The advantage of the rail and guide block system is that the force of the probe assembly on the material under test is constant: the weight of the moving parts (for example the measurement head 1202, the removable mount 1204 and the rail 1206).

FIG. 13 shows a previous embodiment of this invention. The position of the test probe 1302 relative to the reference probe 1304 can be coarsely adjusted by screwing the threaded Luer adaptor 1306 into or out of the frame arm 1308. Fine adjustment comes from turning the screw 1310 with the knob 1312. The frame arm 1308 is held against the tip of the screw 1310 by a spring 1314. In this embodiment the force and motion are generated by a transducer 1316 (Clark Tactile Sound Transducer, U.S. Pat. No. 5,473,700) that consists of two dome shaped disks that are joined at their edges. One supports a voice coil and the other supports a magnet struc-

ture. This figure illustrates that the force generator of this invention is not restricted to just the type of voice coil system shown in the other figures. Other alternatives for a force generator have been shown in FIG. **13** of Methods and Instruments for Assessing Bone Fracture Risk U.S. patent application Ser. No. 11,417,494). This embodiment used a load cell **1318** and an optical position detector **1320**.

FIGS. **14**A and **14**B show two embodiments of this invention. These embodiments are based on a commercially available compact positioning system (VCS-10 Voice Coil Linear Stage from Equipment Solutions, Inc.) In the left embodiment the test probe **1402** is held in a mounting pin **1404** which attaches magnetically to a permanent magnet **1406** that is attached to a shaft **1408** on which is mounted an arm **1410** whose motion is detected by the optical position detector **1412**. The shaft **1408** continues to a load cell **1414** and then to a support block **1416** that is screwed to the movable platform **1418** of a one axis stage with a guide block **1420** under the platform **1418**. The force and motion are generated by a voice coil in magnet structure **1424**. In the right embodiment a shaft **1426** is directly mounted in support block **1416**. In this case the force is monitored as proportional to the current to the voice coil **1428**. In practice this is very close to being an accurate proportionality. If necessary, however, it can be corrected with a correction factor of the moving mass times the acceleration. For example, for a moving mass of 0.1 kg and a maximum acceleration of 100 microns in ten milliseconds, the maximum force correction would be of order) 0.1 kg×100 microns/(0.01 sec)^2=0.1 Newton. Since the embodiment in FIG. **14**B does not have the compliance of the load cell **1414** to deal with, the motion of the test probe can be monitored using the built in position detector **1430** in the VCS-10 Voice Coil Linear Stage from Equipment Solutions, Inc. Alternately, higher position resolution can be achieved with a high resolution LVDT (for example Measurement Specialties MHR 025) or other supplemental distance detectors such as capacitance sensors, optical beam deflection detectors, or laser interferometers to measure the motion of the movable platform **1418** and thus the test probe **1402** relative to the reference probe **1432**, which is stationary relative to the guide block **1420**, which is attached to the case **1434** on which the mount **1436** for the reference probe is attached. Thus the body of the LVDT or other distance detector would be fastened to the case **1434** and the core of the LVDT would be attached to the test probe.

This commercial unit can also be used in feedback mode to run the invention with position control. In this case, for example, the force needed to indent the material under test to a fixed maximum depth could be monitored as a function of cycle number. This was not, however, our preferred embodiment because the force and position noise with the VCS-10 Voice Coil Linear Stage and the SCA824 Linear Servo Controller were much larger than in our preferred embodiment. We believe that some of the problem was due to friction in the one axis stage. Feedback can be more easily used with the much smaller friction from flexures such as in the other embodiment shown in this document. We note that feedback control could also be used to run in a force controlled mode.

FIG. **15** shows another previous embodiment of this invention. In this embodiment the flexure support of the voice coil **1502** in the magnet structure **1504** is provided by two wires. The upper wire **1506** attaches to the shaft **1508** with a cylindrical block **1510**. The outer ends of the upper wire **1506** are attached to blocks **1512** which are, in turn, mounted on flexures **1514** which are, in turn, mounted, with blocks **1516** to the support shell **1518**. The lower wire **1520** attaches to the shaft **1508** with blocks **1522**. These blocks **1522** are mounted

via flexures **1524** as shown above (as **1514**) but rotated 90 degrees around the axis of the lower wire **1520** so the flexures are not as visible as above. These flexures **1524** are mounted on a movable stage **1526** that slides on two rods **1528** and **1530**. This movable stage can be moved by turning the knob **1532** which turns the screw **1534** which connects the movable stage **1526** to frame element **1536** which is held stationary relative to the magnet structure **1504** and the rods **1528** and **1530**. Thus turning the knob **1532** lowers the shaft **1508** and the test probe **1538** relative to the reference probe **1540**. In this embodiment the force sensor **1542** is again a load cell. The position sensor **1544** was optical. A capacitance sensor for position could also be used in this and other embodiments.

FIG. **16**A shows the force measurement for a cyclic indentation cycle test on PMMA taken with the invention as described in FIG. **13**. The corresponding distance measurement over the same set of indentation cycles is shown in FIG. **16**B. FIG. **16**C shows a single indent-retract cycle on PMMA taken with the invention as described in FIG. **15**. The loading cycle consists of an indentation at a fixed rate of voltage drive increase to the force generator, a pause at fixed voltage drive to the force generator, and a retraction at a fixed rate of voltage drive decrease to the force generator.

FIG. **17**A and FIG. **17**B show respectively the measured maximum indentation distance and maximum force after pausing at the maximum drive to the force generator. The pause is included to reduce the effect of viscoelasticity on measurements of the retraction slope and thus the increase the accuracy of the measured elastic modulus. FIG. **17**C shows a linear fit to the initial part of the retraction curve, called the retraction slope, which may be used as a material characterization parameter, or in subsequent analysis to determine the elastic modulus of the sample. FIG. **17**D shows the first and last indent-retract cycles for a series of several indentations taken with the invention as described in FIG. **15**, plotted together for comparison purposes. Change in any measured property over a series of indentation cycles may be measured, as illustrated in FIG. **17**E. The change in maximum force is measured between the first and last indentation cycles over a series of 30 cycles. FIG. **17**F shows the measurement of work during the indent-retract cycle that may be used to characterize a sample. The area beneath the loading and pause cycle is quantified as the work of indentation. The elastic energy recovery is defined as the area beneath the retraction curve. The difference between the work of indentation and the elastic recovery is defined as the energy dissipated in the indent-retract cycle.

FIG. **18** (prior art) shows the measured parameters that are pertinent to the measurement of elastic modulus and hardness in the invention. The variables and equations used in the calculation are listed. The analysis method used is that of Oliver and Pharr (ref. W. C. Oliver and G. M. Pharr. Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology. J. Mater. Res. 19 (2004), 3. (review article)).

The operation of the invention is aided by computer interfacing. FIG. **19** shows a screenshot of the Labview program used to run the invention. The force and distance measurements are collected and plotted both versus time and as a force versus distance graph in real-time. There are several controllable parameters to alter the indentation protocol, including: indentation frequency, indentation amplitude, number of indent cycles, and the half-angle of the conical indenter, called the probe half angle.

Automated data analysis upon completion of the indentation cycles is achieved through the computer interface. A screenshot of the current data analysis interface is shown in

FIG. **20**. The main analysis screen shows several measured quantities that may be used to characterize a material, as well as the raw force and distance data, the identified transition points in each indent-retract cycle, and a comparison of the first and last indentation curves for multiple-cycle testing. FIG. **21**, FIG. **22** and FIG. **23** show additional analysis screen-shots of the change in the measured energy dissipated, the change in maximum adhesion force during retraction, and the change in both elastic modulus and hardness as a function of time through the cyclical test.

FIG. **24** shows a generalized measurement head for this invention. The test probe **2402** consisting of a shaft **2404** and a sharp tip (often a diamond) **2406** is attached to a shaft **2408** which is, in turn, connected to an optional torque and angular displacement sensor **2410** then to an optional torque genera-tor **2412**, then to an optional linear displacement sensor **2414**, then to an optional force sensor **2416**, and finally to an optional force generator **2418**. The reference probe **2420** is connected to the housing **2422** that holds the transducers and generators. The housing **2422** could be supported and posi-tioned on the sample under test by a support such as those drawn in FIG. **12**. The optional torque and angular displace-ment sensor **2410** together with the optional torque generator **2412** can be used to measure friction with test probes such as **1012**, **1022** and **1026** (FIG. **10**) or the torque necessary to screw a test probe like **1028** (FIG. **10**) in or out of a material under test. This might, for example, be useful in determining whether a patient's bone is suitable for holding screws for mounting orthopedic appliances. With the optional force sen-sor **2416** and the optional force generator **2418** the force to pull out a screw could be measured as a test of bone quality.

Although the present invention has been described in con-nection with the preferred embodiments, it is to be understood that modifications and variations may be utilized without departing from the principles and scope of the invention, as those skilled in the art will readily understand. Accordingly, such modifications may be practiced within the scope of the following claims.

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The invention claimed is:

1. An instrument for characterizing a material comprising:
   a housing;
   a test probe and a reference probe aligned in the housing, the test probe constructed for insertion into the material to indent the material;
   a force generator;
   a flexure configured to guide the motion of the force gen-erator; and
   a force sensor operatively coupled to the test probe for determining a force versus distance parameter by mea-suring the force needed to insert the test probe a prede-termined distance or as a function of distance into the material.

2. The instrument of claim **1**, wherein the force generator comprises a moving coil disposed in a magnetic field assem-bly.

3. The instrument of claim **1**, wherein the reference probe is in the form of a sheath in which the test probe is disposed, the distal end of the reference probe being proximal to a tip of the test probe.

4. The instrument of claim **1**, wherein the flexure comprises a first, inner membrane and a second, outer membrane con-nected to the first, inner membrane.

5. The instrument of claim **4**, wherein the first, inner mem-brane is connected to the second, outer membrane via a ring.

6. The instrument of claim **1**, further comprising a distance sensor.

7. The instrument of claim **6**, wherein the distance sensor comprises a linear variable differential transformer.

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